CHYMISTRY AND CRUCIBLES IN THE RENAISSANCE LABORATORY: AN ARCHAEOMETRIC AND HISTORICAL STUDY

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Abstract

This thesis studies crucibles at the interface of alchemy, chemistry and metallurgy. For this, a critical literature review is combined with the analytical study of a range of archaeological remains. The main case study is that of Oberstockstall (Austria), the most comprehensive Renaissance laboratory ever recovered, but samples from other sites in Germany, Portugal, England and the United States of America are also examined. The analytical techniques employed are optical microscopy, energy dispersive X-ray fluorescence (ED-XRF) and scanning electron microscopy energy dispersive spectrometry (SEM-EDS).

A substantially revised historical framework is defined, with a stress on the need to avoid applying present-day labels such as 'alchemy', 'chemistry' and 'metallurgy' to Renaissance laboratory activities, using 'chymistry' instead as a generic and historically more appropriate term. This is particularly relevant as regards the operations of metallurgical analysis or 'fire assay'. Furthermore, the scientific archaeology of alchemy and chemistry is presented as a new research avenue, with well-defined epistemology and applications.

The scientific analyses, in combination with contemporary written information, allow a comparative characterisation of the formal and material properties, as well as the performance characteristics, of the high-temperature ceramics used in Oberstockstall.

From a wider perspective, it is established that dark, usually graphitic, crucibles produced in Bavaria and surrounding regions, competed with the historically better-known sand-tempered, bright Hessian wares, and that both of these high-quality crucibles co-existed with *ad hoc* productions in a variety of contexts of utilisation. The manufacture, appearance and technical standard of the different vessel types are addressed within the wider sociotechnical system. A preliminary approach is presented to the reasons behind choices in the production and consumption of technical ceramics, with an emphasis on past perceptions of different materials.

Finally, further directions for the study of pyrotechnological laboratories are suggested.

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George Eliot (Mary Ann Evans) (1819-1880)

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1. General introduction

"Take interest, I implore you, in those sacred dwellings which one designates by the expressive term: laboratories. Demand that they be multiplied, that they be adorned. These are the temples of the future, temples of well being and happiness; there it is that humanity grows greater, stronger, better."
Louis Pasteur (1822-1895)

The 16th century constitutes one of the most crucial periods in the history of Europe. This relevance is given by several processes developing in the Renaissance which, after much struggle, would lead to the modern political configuration of Europe but also, most importantly, to dramatic changes in the way human beings saw themselves and their surrounding world. These new ideas and attitudes would eventually become the accepted 'common sense' to remain unquestioned and unchallenged in its principles until recent decades. In order to understand this complex and changing Renaissance reality, some facets of particular relevance for this study may be highlighted. Firstly, the 16th century witnesses a new practical input in natural sciences, together with an acme of the quest for the transmutation of metals and the philosopher's stone. Secondly – and equally important –, the century starts with a heyday of mining and metallurgy in Central Europe, both in technological and financial dimensions, before entering a phase of deep crisis. On the whole, this period is characterised by an enthusiasm for erudition and a humanistic interest in codifying knowledge that suitably coincide with the spread of the printing press. And consequently, there is a surge in the experimentation with chemical substances, together with new theories of matter that appear expounded and debated in a plethora of writings.

As practical experiments gave rise to new notions about the natural world and *vice versa*, people active in chemical and metallurgical activities increasingly required a set of specialised and standardised instruments and techniques. These should allow the conduct of specific reactions with a developing concern over accurate measurements and reproducibility. As a result, the laboratory emerged as a specialised workplace for

chemical experiments. This step entailed the beginning of a very significant transformation: before that period, "one possessed *scientia*"; after that, "one did science" (Hannaway 1986: 586). The laboratory instrument, as the intersection between theory and experience, the crucible where pure and applied knowledge interacted, facilitated this change – and it remains as the physical illustration of it, with almost iconic value.

This thesis takes as a starting point the archaeological remains of the most comprehensive Renaissance laboratory ever recovered: that from Castle Oberstockstall in Kirchberg am Wagram, Lower Austria. The assemblage, closely dated to the second half of the 16th century, comprises complete artefacts and fragments of about eight hundred pieces of equipment including, amongst others, triangular crucibles, scorifiers, bone ash cupels, alembics, aludels, cucurbits, adopters, receivers and other chemical equipment of ceramic and glass, as well as metal and slag remains, furnace bricks and other artefacts of leather, textile and bone (Soukup and Mayer 1997; von Osten 1998). All in all, this is believed to be a state-of-the-art 16th-century laboratory, and thus an extraordinary *source of information.* However, at the same time, Oberstockstall shall serve in this thesis as a *source of inspiration.* The original aim of this thesis was a reconstruction of the intra-history of this specific Renaissance laboratory. However, the research questions that this topic evoked requred a wider consideration of the developing range of laboratory equipment and techniques, which led the project to embrace new case studies across the world and respond to broader questions. As a result, this work presents a reference study of the manufacture, distribution, use and performance of specialised ceramics used for chemical operations within an environment of fundamentally changing perceptions of the nature of elements and chemical reactions. It is the author's purpose to contribute to the history of science and technology as much as to the archaeology of the Modern Era. More specific aims and objectives are detailed in the next section.

1.1. Aims and objectives

This thesis endeavours to respond to some precise enquiries, specified in section 1.1.1. In addition, it is expected to add significantly to a variety of broader research topics, as indicated in section 1.1.2. These aims and objectives outline the originality of this thesis and its value in the wider research framework, thereby justifying the work.

Besides, the results and discussion of the theoretical issues involved shall serve to argue the need for further research along similar lines.

1.1.1. Aims

a) Comparative characterisation of the technical ceramics used for chemical operations in the Oberstockstall laboratory, with a special focus on those used for high-temperature reactions. Study of their manufacture and definition of fabric types that might be related to different producers.

b) Analysis of the technical quality of the different formal types and fabrics documented in the laboratory ceramics, and assessment of their behaviour under their various working conditions. Appraisal of the knowledge and purpose exhibited in the selection and processing of different raw materials.

c) Preliminary consideration and assessment of the practical use of these instruments, and the range of reactions carried out in the laboratory.

d) Identification of technological choices in the manufacture, acquisition and use of the laboratory equipment. Exploration of the reasons behind those choices, with a consideration of the relevant socio-cultural context.

e) Study of the main types of crucibles produced and traded in post-medieval Europe, their manufacture, original appearance, material properties and geographic distribution.

1.1.2. Objectives

a) Definition of a methodology and epistemology for the scientific analysis and interpretation of technical ceramics from archaeological contexts, aimed at answering specific research questions and joining together interests traditionally divided amongst different specialists such as ceramicists, archaeometallurgists or historians.

b) Justification of the need and value of science-assisted archaeology as a new, complementary approach to the history of science.

c) Definition of a sound theoretical framework for the scientific archaeology of Renaissance science and technology, some of whose concepts may be of use for a wider spectrum of studies of ancient technologies, particularly those involving the combination of archaeological and written sources.

d) Clarification of the role and practice of alchemy/chemistry in Renaissance Europe, and their relationship with metallurgy. Discussion of the status of fire assay

within this framework, and in the development of modern metallurgy and analytical chemistry.

e) Exploration of the material, social and cultural parameters surrounding the production and consumption of crucibles in the post-medieval world. Consideration of the crucible as a socially embedded constituent of material culture.

f) Approach to the changing perception and understanding of materials current in the Renaissance.

1.2. Princes, patrons, miners and the philosopher's stone: geographic and historical context

In the 16th century, what was left of the Holy Roman Empire covered the area extending from the Rhineland and Switzerland in the West, through Hesse and Bohemia to include Silesia, Moravia and Lower Austria in the East, from the North Sea in the North to Tuscany in the South (fig. 1). The Austrian House of Habsburg dominated the post of the emperor. However, the political power was very fragmented amongst the rulers of the more than three hundred feudal, ecclesiastical and urban territories in which the Empire was divided. These sovereigns sat in the *Reichstag* – the Imperial Diet – and could control the emperor, who was almost just as powerful as any other of them. In this context, the Lutheran Reformation prompted an exacerbation of the political disputes under the disguise of religious discord. When the emperor Charles V abdicated in favour of his brother Ferdinand in 1558, the 'Holy Roman Empire' seemed just an ironic denomination for an heterogeneous land housing fierce dissension between Catholics and Protestants, struggling to keep Rome within its domain, and lacking an absolute governor (Koenigsberger *et al.* 1989; Nicholas 1999).

Figure 1. Map of Central Europe in 1555, with an indication of the most relevant places to this thesis (after Roberts 1991: 149, modified).

The political and religious conflict was not, however, an obstacle for economic and cultural developments. During the first half of the century, some areas of Central Europe reinforced their role as key centres of trade and industry based mostly on the export of linen textiles and ceramics, as well as the production of metals. Between 1460 and 1530, the production of silver, lead, copper and other metals increased impressively, up to five times over (Neff 1987). This boom encouraged technical solutions – exploitation of deeper and/or poorer veins, water removal… – but also entailed organisational changes, since capital and labour were increasingly separated, and hence wealthy laymen became mining patrons. Just as the mining enterprise developed, so did the specialisation of labour. Progressively, competition with the steady arrival of relatively cheaper silver from America put an end to this heyday, and European metallurgists had to make virtue of necessity. During the second half of the 16th century, capitalist mining and metal production continued in Europe, now on the strength of specialists seeking the most efficient methods of assaying as well as metal extraction (Neff 1987; Long 1991; Roberts 1991).

Even more important was the intellectual atmosphere and the current understanding of nature. In 1500, the dominant philosophical influence in Europe was a mixture of Christian and Aristotelian thought, as synthesised by Saint Thomas Aquinas in the 13th century (Jordan 1992; Elders 1997). This comprehensive system affected the worldview as much as the religious behaviour and the understanding of natural phenomena, and was roughly shared by all sections of society, from the highly educated humanist to the illiterate peasant. Difficult as it may prove for us to understand this intellectual orderliness, for an average 16th-century person this was not philosophy but just 'common sense' or 'received wisdom' beyond question.

In short, for a Renaissance person, everything ultimately consisted of prime matter. The four fundamental qualities of the prime matter were heat, cold, dryness and moisture (or fluidness), which were coupled in two pairs of opposites – hot/cold, dry/wet (or fluid). Accordingly, the four elements were the materialisation of the four pairs of qualities: *fire* was hot and dry, *air* was hot and fluid, *water* was cold and fluid, and *earth* was cold and dry. In each element, one quality was predominant: dryness in earth, cold in water, fluidity in air, heat in fire. Since all the natural elements were made of the same prime matter – they only showed different qualities –, then they could be turned into one another by changing their qualities: thus, for example, fire could become air by means of heat, and water could become earth by means of cold (Holmyard 1968) (fig. 2). This principle applied to everything one could see in the world. If the proportion and combination of the qualities in a reality changed, the fundamental nature of that reality changed as well. Even the human body was a factor of those qualities, manifested in four humours: phlegm, blood, bile, and black bile. In a healthy human, the humours were said to be balanced; illness resulted from a deficiency or excess of one of these, and the role of the physician was to restore the right balance.

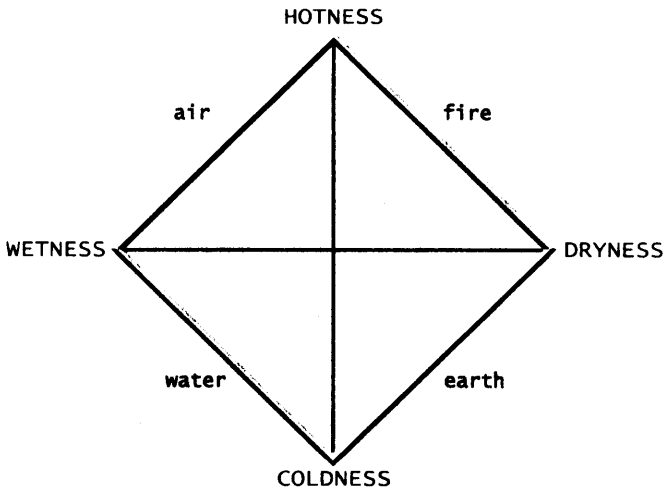


Figure 2. Diagram of Aristotle's four element theory.

Within this mental framework it is not surprising that metallic transmutation was conceived by many as something feasible. In spite of the limitation posed by the fact that humans could not control the influence exerted by heavenly bodies in the natural transformation of matter, it was worthwhile trying to replicate nature. In the same way as water allowed the transformation of a seed into a tree, or just as heat transformed crumbly iron ore in solid metal, why not think that lead could be transformed into gold by altering its qualities?

Europe lacked order and equilibrium, and the philosopher's stone was the appealing counterpoint of this. As the purest manifestation of the prime matter, it represented the perfect equilibrium of qualities and constituted the key ingredient allowing transmutation. This was understood by the many wealthy people that increasingly became patrons and sponsors of art and science, not only of the 'Great Work' towards the philosopher's stone but also of astronomy, mathematics, philosophy, geography... and all the other intertwined branches of the tree of knowledge. The most infamous representative of this extent was to be the Holy Roman Emperor Rudolf II, who ruled between 1576 and 1611 and made the new court in Prague a centre of knowledge and research aimed at fulfilling his ambitions and satisfying his eccentric – if not mad – taste (Evans 1997). There were many more such patrons, nonetheless. Exploring the secrets of nature advanced the knowledge of the cosmic equilibrium, and this knowledge was the key to power and the order of things. The discoveries of this research would end up undermining the very principles that had encouraged it, and by 1700 we will have a much more diverse and socially uneven worldview and understanding of nature (Wolf 1950; Wightman 1962; Smith 1972; Debus 1978; Knight 1992; Henry 2002).

Another important dimension of the Renaissance was the increasing mobility of people across Europe and beyond. Whilst the political configuration of Europe evolved towards the constitution of separate nation-states, these newly created borders were often crossed by growing numbers of craftsmen, entrepreneurs and intellectuals seeking convenient markets, patrons or instructors. This facilitated the transfer and exchange of goods, customs, knowledge and ideas within this atmosphere of confidence on human nature, and ultimately led to the discovery and colonisation of the New World (Belfanti 2004).

Although the origins of the Renaissance may be traced back to 13th-century Italy, the major developments of this movement did not emerge in Central and Northern

Europe until the late 15th century. Depending on the focus, scholars tend to propose different time frames and causes for the changes leading to the so-called 'Scientific Revolution', which will not be discussed here. It may suffice to say that, between 1450 and 1700, many factors such as an epoch willingness to experiment, question and explore the boundaries of knowledge, capitalist investments in technological advance for the sake of economic profit, great maritime explorations, warfare and medicine needs, and the rediscovery of classical texts as coming from a 'Golden Age', altogether led to dramatic changes in the human approaches to their environment. Some consequences of this fundamental transformation bear a special relevance here, namely an increasing challenge to the traditional principle of authority and a rising input in experimentation.

This is the context of the advances in mining and assaying technology, of the philosopher's stone craze, and of pioneer investigations into nature. This is also the context that favoured the development of modern science in general, and that of chemistry in particular. Lastly, this is the wider historical context of the archaeological remains addressed here. The assemblage from Oberstockstall will serve as a focal point to study theories and practices as revealed in this laboratory, but the scope will be widened to embrace other relevant case studies from within the Holy Roman Empire, stretching out to Britain and Portugal, and even reaching early colonial America. By blending the archaeological study with the analysis of the historical context, this thesis strives to ultimately advance our knowledge of Renaissance peoples.

1.3. Structure of this thesis

This thesis starts with a critical approach to the existing knowledge and assumptions regarding the scope and interaction of Renaissance alchemy, chemistry and metallurgy; special attention is paid to fire assay as a repertoire of reactions widely documented in Oberstockstall and elsewhere, which would bear a crucial repercussion in the development of modern experimental science (chapter 2). Chapter 3 builds on this foundation to present an outline of more specific problems of traditional, text-based approaches to alchemy and chemistry, and discussion of how the scientific archaeology of laboratory remains may help overcome such limitations. Also in this chapter, the role of the instrument as an active constituent of the scientific endeavour, linking theory and practice, is addressed. Some theoretical and epistemological concepts are introduced, borrowed from material culture studies and sociohistorical approaches

to technology. A wider framework is thus defined to contextualise scientific studies of laboratory remains, and generally to facilitate communication amongst historians, archaeologists and scientists. The rest of the thesis constitutes a contribution to this picture, but it is hoped that the theoretical background shall be of use for future research.

Chapter 4 provides an introduction to the main case studies considered in this thesis, with details of archaeological contexts, assemblages and previous work carried out with them. In chapter 5, the different analytical techniques employed are briefly introduced, and their choice is justified on the basis of several criteria. Analytical protocols are detailed to allow for replication of data or subsequent studies coherent with this.

Chapter 6 contains the bulk of the analytical results and discussion pertaining to the laboratory equipment from Oberstockstall, tied with an examination of relevant written sources. This provides an approach to the manufacture, material properties and performance of the various technical wares, together with an initial appraisal of their uses. The technical knowledge and understanding of the different elements current in this laboratory, as documented in the archaeological remains, are discussed.

From chapter 7, this thesis concentrates on the crucible as the foremost instrument used for high-temperature operations, but the temporal and geographic scope is widened to embrace different case studies from post-medieval Europe and beyond. Altogether, a preliminary attempt is made at reconstructing the manufacture, distribution and consumption of crucibles, which will set the groundwork and raise several questions for further work. Firstly, the little-known black, smooth crucibles produced in Bavaria and surrounding regions are addressed (chapter 7). Secondly, the analysis moves on to the famous bright, pimply crucibles from Hesse (chapter 8). The picture is completed with the study of some crucibles produced outside these major regions (chapter 9). In all cases, historical, archaeological and analytical information are systematically combined and confronted, and the pertinence of integrated approaches becomes apparent. As a result of this multidisciplinary strategy, the provenance of most of the crucibles studied in this thesis is identified for the first time, and their technical properties are discussed in detail.

In chapter 10, the information obtained up to that point is joined together with that from further historical literature and anthropological approaches to technology. A more comprehensive picture is presented of the *chaîne opératoire* of the crucibles, placing

special emphasis on the perception of different elements and the choices made by post-medieval peoples, both on the production and the consumption sides. The success of the more widespread crucible types is examined within the broader sociotechnical system, and an initial exploration is presented of the possible reasons leading to various technological choices in the manufacture, acquisition and use of these vessels at different times. Many of these issues are revealed as intrinsic and informative aspects of early technology and science that had so far been overlooked.

Chapter 11 presents the main conclusions of this thesis as well as an outline of some avenues for future work stemming from it.

The appendices contain analytical results of individual samples. Appendix 1 is comprised of tables with comprehensive chemical compositions of the ceramic matrices of all the samples discussed in the text, for which only averages are given within the body of the thesis. Appendix 2 presents more detailed micrographs and analytical details of some samples with relevant metallurgical residues.

2. Alchemy, chemistry and metallurgy: fire assay in Renaissance Europe

"Truth, like gold, is to be obtained not by its growth, but by washing away from it all that is not gold."
Leo Tolstoy (1828 - 1910)

"My Lord, facts are like cows. If you look them in the face hard enough, they generally run away."
Dorothy L. Sayers (1893-1957)

2.1. Introduction

Fire assay is the quantitative determination of the presence of specific metals in a given substance, using high-temperature chemical processes similar to those in a metallurgical furnace. The most prominent fire assay is the analysis for gold and silver. Normally, this involves first liberating the precious metals from their original host matrix, then transferring them into a quantity of lead metal that acts as a collector, before then separating these noble metals from the lead utilising their different oxygen affinity. These three steps, *i.e.* concentrating the metallic components, collecting the noble metals and cupellation, are represented archaeologically by a series of rather specialised and diagnostic finds such as certain crucible types and, above all, the unique bone ash cupels. The same set of tools is in use to this day, albeit now made from different materials.

The historical practice of fire assay has been addressed from different points of view. Traditionally, historians of mining and metallurgy have focussed on the various metallurgical treatises and handbooks written during the Renaissance as a main source of information. Thanks to this corpus of works we have learned, for example, the important role of the numerous assayers who worked in central European mines, specialised on analysing the richness of ore veins and controlling the composition of the metals produced and traded (e.g. Sisco and Smith 1949; 1951; Smith and Forbes 1969). The scientific study of archaeological remains of assays has added valuable information regarding the particulars of the tools, raw materials and techniques utilised, normally

juxtaposing them to those described in metallurgical written sources. Whilst analyses of this sort used to be concerned with the mere adscription of some given finds as assay remains, more recent archaeometric work has given increasing attention to explaining the chemical reactions undertaken (e.g. Pittioni 1975; 1978; 1985; Bayley and Barclay 1990; Bayley 1991a; 1991b; 1992; Bayley *et al.* 1991; Eckstein *et al.* 1994; Bayley and Eckstein 1997; Rehren 1996a; 1997; 1998; 2002a; Schifer 1998; Martinón-Torres 2003). Finally, historians of alchemy and chemistry have started to emphasise the importance of fire assay in the development of modern science and use texts by early alchemists and chemists to address this issue (Halleux 1986; Szabadváry 1992; Newman 2000; Newman and Principe 2002: 35-91).

Notwithstanding this array of research lines, by and large, the present-day compartmentalising of academia has resulted in a lack of cross-disciplinary approaches. Historians of mining and archaeometallurgists have constrained their focus to assemblages from what is loosely termed early metallurgy, whilst historians of alchemy and chemistry have tended to concentrate on early al/chemists and textual evidence for the practice of fire assay in their laboratories. For the former researchers, alchemy is often disregarded as an overly theoretical endeavour bearing little in common with the practical, experience-based craft. For the latter, metallurgists are sometimes overlooked as mere craftsmen practising chemical reactions but not caring about the scientific principles behind their operations. Consequently, both groups of scholars have defined different research objectives and normally approach them without taking into account the work carried out in the other field. Although scholars such as Herbert and Lou Hoover (1950) in their translation of Agricola's *De Re Metallica*, and Cyril Stanley Smith in several co-authored translations and publications (Smith and Forbes 1969; Hawthorne and Smith 1979; Smith and Gnudi 1990), have from the beginning emphasised the importance of cross-disciplinary studies, including both chemical and metallurgical expertise, only little of this is reflected in current work.

Recently, the study and interpretation of certain groups of archaeological finds from contexts without an *a priori* clear connection to either metallurgy or chemistry have required a consideration of both metallurgical texts and al/chemical activities, thus indicating the need for more comprehensive approaches (Rehren 1996a; Kamber *et al.* 1998; Rehren 2002a). The practice of fire assay as an analytical tool is equally relevant to chemistry, extractive metallurgy and quality control in coin production; even the small-scale recycling and refining of jewellery employs the same operations. As a result, this

seemingly diagnostic archaeological evidence is indeed highly indicative of fire assay, but does not in itself enable identification of the immediate technological context within which these analyses were performed. In some instances, the archaeological context will be sufficient to establish the technological context, as is the case of the finds from the Archbishop's Palace in Trondheim (Norway) (McLees 1996; Saunders 2001) or the Tower of London (Bayley 1996), both of which are clearly related to the assay of coinage, and the finds from the Frobisher Expedition on Kodlunarn Island (Canada) (Auger *et al.* 1995; Unglik 2000; Beaudoin and Auger 2004) or the abandoned mining town of Münster in the Schwarzwald (Germany) (Schifer 1998), both of which are clearly aimed at the analyses of ores to guide subsequent mining and smelting activities. Other cases are much less obvious in their interpretation, particularly when they are from a habitation context (Rehren 1996a; Kamber *et al.* 1998; Rehren 2002a). The differentiation between chemical and metallurgical fire assay requires a much wider investigative approach, and may still prove futile.

This thesis, taking Oberstockstall as the main case in point, sets out to attempt a more holistic reappraisal of fire assay. The assemblage, recovered beneath the sacristy of a church at a manor house, contains fragments of almost three hundred triangular crucibles, fifty scorifiers and one hundred bone ash cupels, making it clear that fire assays were conducted there on a frequent basis (fig. 3). In addition, fragments of a range of chemical equipment such as alembics, aludels, cucurbits, adopters, receivers and other vessels of ceramic and glass are present, together with minerals

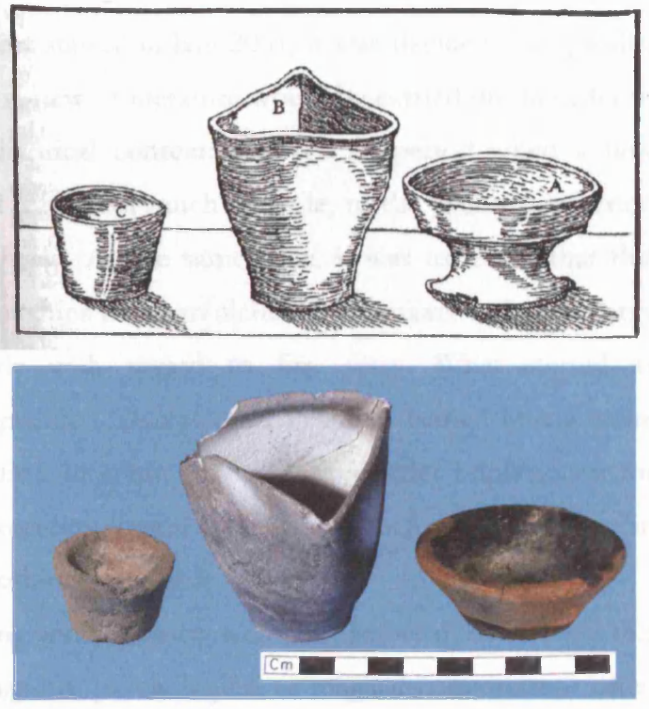


Figure 3. Top, the basic fire assay equipment as depicted by Agricola (Hoover and Hoover 1950: 229), from left to right, a bone ash cupel, a triangular crucible and a scorifier. Bottom, the basic set of tools as identified in the Oberstockstall assemblage (scalebar 10 cm). This parallel was first identified by Thilo Rehren.

and metallurgical debris[1]. All this raised the question of whether this was the workshop of a mineral assayer, the laboratory of an early alchemist or chemist, or whether both fire assays *and* al/chemical operations had been undertaken there.

The first comprehensive study of this assemblage was carried out by the director of the excavations, Sigrid von Osten, in her 1992 doctoral thesis, later expanded and published in 1998. She presented a thorough catalogue of finds and, based on some contemporary written sources, offered a preliminary interpretation of the functions of most instruments. When reviewing von Osten's thesis, Sperl (1994) already posed this crucial question: was this an assayer's workshop or an alchemist's laboratory? Later on, Soukup and Mayer (1997) broadened the scope of comparative written sources and provided more details as to the chemical reactions possibly undertaken in the different wares. They also announced a programme of scientific analyses of the assemblage (Soukup and von Osten 1992; Soukup and Mayer 1997: 48-49), however only a few results appear cursorily outlined in their 1997 volume and, as far as I am aware, no more analytical results have been published. In any case, that basic question remained unaddressed.

When the present PhD project started in late 2001, it was decided that, parallel to the scientific analysis, a widescale review of literature would be carried out in order to place the activity into its larger historical context: this crucial period when a new emphasis on experimentation would lead, with much struggle, to the eventual rejection of the traditional Aristotelian worldview. At the same time, it was expected that this study would shed light on the relationships between alchemy, chemistry and metallurgy in Renaissance Europe, particularly with regard to fire assay. What started as background research to answer a specific archaeological problem turned into a more substantial venture, with the realisation of some issues bearing wider implications for the study of laboratory-based pyrotechnological operations such as fire assay in Renaissance Europe. Two main hypotheses emerged:

a) In the Renaissance, alchemy and chemistry were *not* distinct domains, and this inclusive field did *not* convey the religious, psychological or magical connotations often attached today to the pursuit of metallic transmutation.

b) Fire assays were al/chemical as much as metallurgical operations and, as far as assays are concerned, it is often impossible – and inappropriate – to separate one sphere

[1] An introduction to the archaeological context and the assemblage is given in section 4.1. Analytical results are presented in chapter 6 and discussed throughout the rest of this thesis.

of activity (alchemy/chemistry) from the other (metallurgy), given their strong intertwining.

These fundamental factors are rarely acknowledged in the literature on the history of science, and are virtually absent in publications on mining history and archaeometallurgy. Their discussion will form the remaining parts of this chapter.

2.2. Renaissance alchemy and Renaissance chemistry

The range of chemical equipment present in the Oberstockstall assemblage suggested that this could be the laboratory of an alchemist or chemist. At first glance, the context of the findings seemed to indicate an 'alchemical' origin: the location of the laboratory in the sacristy could have been where the alchemist would seek divine inspiration in his quest for the philosopher's stone, and therefore appeared quite meaningful. Strikingly, there is a small slot open in the wall of this sacristy that allows a direct view of the altar from the laboratory (fig. 4). Favouring this preliminary interpretation was also the 'alchemical' imagery present in the assemblage, including a tile with a depiction of a half open door typical of later freemasonry (fig. 5). It was tempting to imagine the Renaissance sage fiddling with crucibles and enigmatic books in a

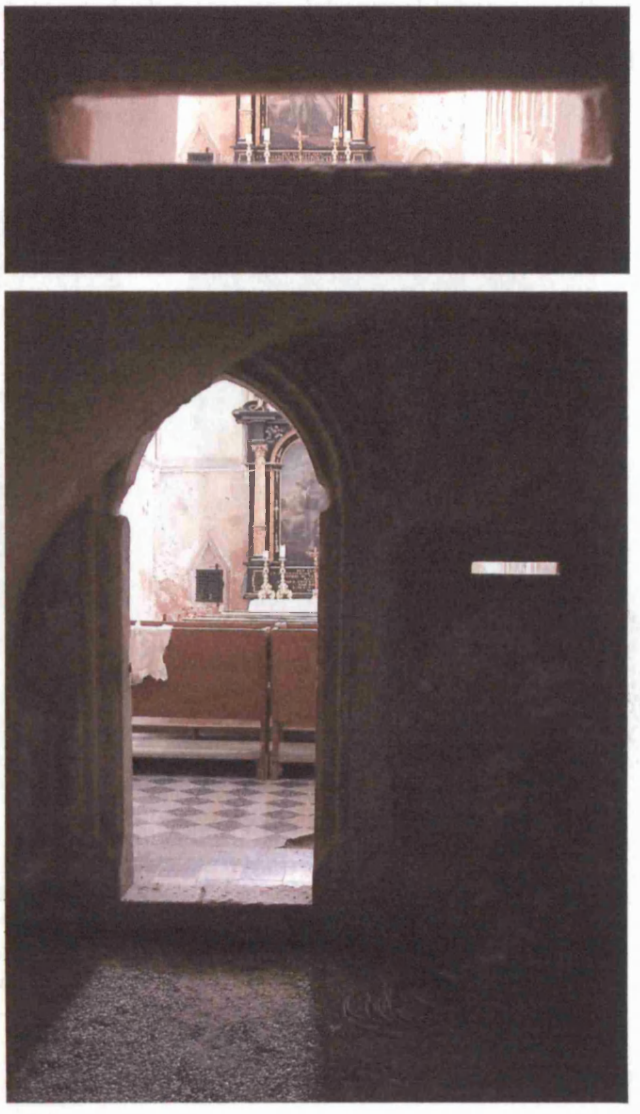


Figure 4. Bottom, image taken from the sacristy and laboratory in Oberstockstall, where a slot in the wall can be noticed. Top, detail of the view of the altar through this opening.

smoky laboratory, aiming for the purification of his soul and personal transmutation while deliberating on the meaning of the philosopher's stone. However, especially as the archaeometric study progressed, the technical quality and degree of specialisation shown by the different utensils and debris suggested that this was rather a 'modern', scientific laboratory, where specialised chemical operations were carried out systematically. This dichotomy seemed of the greatest importance. In accordance with the bulk of the secondary literature on alchemy and chemistry, a crucial assumption had to be made: should our study prove that the Oberstockstall laboratory was that of a chemist, then this would constitute a key assemblage to document some particulars in the development of present-day science; if, instead, this was an alchemical laboratory, then the remains could provide information regarding the rituals and obscure arts practised by the adepts whilst invoking supernatural powers.

Figure 5. Left picture: cover engraving of Heinrich Khunrath's *Amphitheatrum Sapientiae Aeternae* (1595)[2]. On the left, mysticism; on the right, experiment; in the foreground, musical instruments representing the necessary harmony between those two; in the background, a half open door, the path to knowledge, which would become an icon of freemasonry. Right picture: a tile with an almost identical door, recovered in Oberstockstall (height of the tile is 325 mm). This parallel was first noted by Sigrid von Osten.

[2] This image is taken from one of the only three copies known, deposited in the Department of Special Collections of the Memorial Library at the University of Wisconsin-Madison, and accessible via the website <http://www.library.wisc.edu/libraries/SpecialCollections/khunrath/index.html> [accessed 10.04.2003].

Were we dealing with alchemy or with chemistry? Were they philosophers or scientists? It turned out that the very phrasing of the research questions was inappropriate.

2.2.1. Alchemy or chemistry? Terminological issues

It is irrelevant to question whether a 16th-century person should be called 'alchemist' or 'chemist' if one realises that such a distinction did not exist at the time concerned. In present-day language, 'alchemy' immediately recalls the idea of the transmutation of metals and gold making – often with many other connotations, as discussed below. 'Chemistry' is, in turn, reserved for the more 'rigorous' discipline concerned with the composition of things. However, that neat definition of alchemy as gold making did not appear until the 18th century, and did not crystallise before it was included in Diderot and d'Alembert's *Encyclopédie*, published in 1753. Before this, the two words were synonymous; when discrimination was made, this was specific to each individual author and based on grounds different from the modern ones. One of the best known examples attesting to this is Andreas Libavius' 1597 volume which, even if entitled *Alchemia*, only devotes a few pages to discuss transmutation, and is today widely recognised as the first modern handbook on chemistry. Biringuccio (1540) talks about *arte alchimica* when discussing the philosopher's stone (Book I) but also when describing sublimation and distillation (Book IX). Agricola (1556), instead, uses the term *chymistas/chymistae*, both when discussing 'bad' alchemists who deceive people, as he does in his Preface, and 'good' ones who developed early assaying methods, as referred to in his Book VII of *De re metallica*. Obviously, these details can only be noticed when working with the primary sources in their original languages, since translators may not have been aware of the importance of these terminological subtleties. There are many other figures to illustrate this linguistic identity of 'alchemy' and 'chemistry', and even examples of single authors using both terms indistinctively (Newman and Principe 1998).

What is the origin of this confusion? Seemingly, a wrong interpretation of the etymology. The prefix *al-* of *alchymia* is nothing but the Arabic definite article added to the Greek *chēmeia* or *chymeia*, probably derived from *cheein*, the word used for smelting. This *al-* was misinterpreted in the 17th century as connoting special excellence. Subsequently, the term *alchemy* was progressively reserved for what was considered the magnificent side of the discipline, *i.e.* the gold making endeavour. Hence "synonyms

became non-synonymous" (Newman and Principe 1998: 41) and, contrary to the present day, higher status was given to 'alchemy' and its quest for the philosopher's stone. Afterwards, as the 'Great Work' was repeatedly proven to be impossible to achieve and increasing numbers of frauds were reported, professionals tended to legitimise their activity by dissociating it from the now less reputable 'alchemy'. In 1685, Sir John Pettus already notes that, in spite of meaning the same as "chimistry", the term "alchimy or alchimist (...) is sometimes taken in an ill sense" (Pettus 1685: 25). Thus the throne was turned upside down, 'chemistry' became the respectable occupation, and the foundations of the modern historiographic mistake were established.

The identity of Renaissance alchemy and chemistry has been pointed out by several historians of science (Goltz 1968; Weyer

Figure 6. David Teniers the Younger (1610-1690), *Alchemist Heating a Pot*. A sympathetic view of the alchemist as a practitioner: a serious researcher in an orderly laboratory (cf. Principe and De Witt 2002: 12).

Figure 7. Charles Meer Webb, *The Search for the Alchemical Formula* (1858). A depiction of the decrepit alchemist suffused in obscurity and mysticism (cf. Principe and De Witt 2002: 34).

1978; Halleux 1979; Rocke 1985; Newman and Principe 1998). It is surprising that many historical and archaeometallurgical studies still address – or simply take for granted – the differences between alchemy and chemistry. Some scholars admit the character of alchemy as a 'primitive' or 'pseudo' science that, despite much obscurity and abstraction, somewhat contributed to the development of modern chemistry (e.g. Partington 1960; Debus 1978; Knight 1992; Levere 2001) (fig. 6). Others diminish the practical side of alchemy and emphasise its connections with magic, astrology, religion or psychology, contrasting it with its contemporaneous chemistry, which is seen as the root of modern science (Eliade 1938; Jung 1939; Eliade 1962; Lange 1965; Burland 1967; Smith 1972; Fabricius 1976; Jung 1980; Beretta 1993) (fig. 7). Another popular position entails disregarding alchemy altogether as a vague craze of the ignorant and the greedy (fig. 8), again dissociating it from 'proper' chemistry. Regardless of their conclusions, all these perspectives suffer from a strong bias. In fact, since 'alchemy' and 'chemistry' did not exist as distinct disciplines in the Renaissance, their tentative differentiation by historians of science is anachronistic and confusing. As aptly contended by Newman and Principe (1998: 36),

> "If one defines alchemy as a non-quantitative, vitalistic or vaguely 'non-scientific' endeavor, and then differentiates source materials into alchemy and chemistry using these criteria, the findings will of course reconfirm the definition and all that accompanies it".

This distinction is set *a priori* and founded on modern conceptions, these parameters being completely alien to 16th-century al/chemists and therefore not valid as a theoretical vantagepoint for scholarly study.

Given that this mistaken segregation between Renaissance 'alchemy' and 'chemistry' is so deeply engrained in the historiography, the use of the archaic term *chymistry* has recently been suggested as a neutral, all-inclusive denomination for both 'alchemy' and 'chemistry' in the period spanning from the Reformation until the end of the 17th century. For earlier periods, the word 'alchemy' would remain valid, as the Latin *chymia* rarely appears before that time. In order to specifically denote the transmutation of base metals into gold and silver, respectively the terms *chrysopoeia* and *argyropoeia* may be used, just as *chemiatria* or *iatrochemistry* may be reserved for the preparation of medicines (Newman and Principe 1998: 41-42). Further, *spagyria* is specifically the separation of a substance into its Essentials (Principe 1998: 9). This terminology avoids

confusions and unfounded presumptions while acknowledging the existence of a single sphere of activity with multiple dimensions. Its use is henceforth adopted in this study and encouraged for others.

Figure 8. Pieter Brueghel the Elder, *The Alchemist* (1558). A view of the greedy alchemist in a chaotic laboratory, about to melt his last coin in the crucible while his wife shows an empty purse (compare to the full purse hanging from the belt of the alchemist in Figure 6), and his foolish assistant bellows some spilled crucibles. Through the window, a view into the future presents the same group, now ruined and begging for charity (cf. Principe and De Witt 2002: 11-12).

2.2.2. Proto-philosopher or proto-scientist? The connotations of chrysopoeia

Like many other historians of science, Cottie Burland (1967: 1) wondered:

> "Was the alchemist a philosopher, deep in a mysterious study where he discovered the secrets of transmutation? Was he just a charlatan involved in a particularly fantastic kind of mumbo-jumbo? Was he simply a forerunner of the scientists of today? Or had he an occult knowledge which we cannot hope to acquire in our materialist environment?"

Similar questions were raised when first addressing the Oberstockstall assemblage, hoping that they would explain the wider context and implications of the archaeological remains. However, once again, the questions had been based on unsubstantiated assumptions.

Whether we call it 'alchemy' or, more correctly, 'chrysopoeia', the idea of the Renaissance sage trying to transmute base metals into gold tends to bear several connotations. These are rooted in the Enlightenment rejection of obscurantism and in the later Romantic disillusionment with Newtonian science that led to a new turn to occultism (Principe and Newman 2001). Nevertheless, what is the historical evidence supporting them? Startlingly, very little – if any. In the following pages, drawing mainly from a seminal article by two historians of science (Principe and Newman 2001), some of the common interpretations of Renaissance transmutational chymistry are examined.

a) Chrysopoeia is related to natural magic, astrology or soothsaying

Many historians and other scholars include transmutation – what they call 'alchemy' – in the catalogue of 'obscure arts' (e.g. Evans 1997). As they appear concealed in secrecy and 'initiatic style', writings and activities related to chrysopoeia have traditionally been related to different types of 'secret knowledge', such as magic and astrology. The reason behind this connection is simple: what was secret and murky in its own time became incomprehensible or meaningless later on and, as a result, anything 'obscure' has been typecast together in later interpretations. In other words, we have created a 'hotchpotch' category with all the past activities that appear hard to understand in the present.

It is time for this easy and erroneous simplification to be avoided. Pursuing metallic transmutation did not necessarily imply magic or astrological practices, not to

mention soothsaying. Of course both realms coexisted in some individuals, as it happened with other disciplines – in the same way as a present-day doctor, or a housewife, or a plumber, may be superstitious. Significantly, the boundaries between Renaissance science and magic were diffuse, therefore some practices that would today be labelled as 'magic' would not surprise any 16th-century 'scientist' (cf. Henry 1990). The case of chrysopoeia was not special in this point, since it matched the current theories of matter, which made transmutation appear feasible. This enterprise, however, could prove very profitable and give power to those unworthy of it, hence the need for suitably encrypting the knowledge gained. There was, however, a long distance between codifying information and practising magic.

b) The gold makers' goals were supramundane

It is very common to believe that the main goals of the gold maker were beyond this world. Widespread understanding of the quest for the philosopher's stone depicts the 'alchemist' as a possessor of a large arcane knowledge and spiritual enlightenment. According to this view, the transmutation or 'healing' of metals would be an allegory of the transmutation of the adept himself, from a mundane, terrestrial state into a more noble, spiritual or divine condition. This allegedly praiseworthy picture of the alchemist has been underpinned by a wealth of scholarly books and popularised in films, tales and novels (fig. 9).

The problem of this interpretation is simple: "even laying aside the more extreme positions, the historical record (...) simply does not countenance it" (Principe and Newman 2001: 397). Even though many chymical texts include fairly extensive imprecations to divinities, exhortations to morality and expressions of piety, there is no evidence to support the idea that their works aimed at anything other than the material gold. The religious sensitivity of the Renaissance was greater than that of later periods, which somewhat led to overstating the input of religion in the 'Great Work' when reading ancient chrysopoetic books from more secular contexts – an ahistorical interpretation. Still

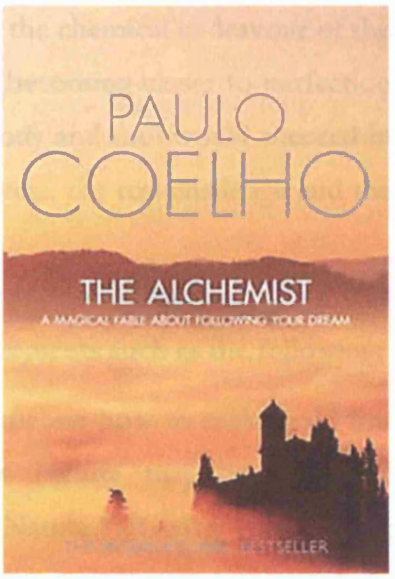


Figure 9. Cover of Paulo Coelho's popular novel *The Alchemist*, the story of a boy who, guided by an alchemist, learns how to overcome his fears and follow his dreams.

during the Renaissance, some unskilled adepts, unable to decipher the texts, may have thought that the key to the philosopher's stone was spiritual rather than practical. This pious affection of the time, the emphasis on secrecy and even the use of religious allegories to illustrate and adorn chymical works led to the identification of the gold maker as a sort of mystic. The actual use of the rhetoric of transmutation as a didactic metaphor in the religious literature also contributed to this confusion. As Gareth Roberts has noted,

> "Alchemical discourse was also suffused with the language of Christianity, its ethics, scriptures, liturgy and doctrines. [Religious images] were central to the Christian doctrine and also to the mystery religions, both of which provided *cultural contexts* for the earliest alchemical texts." (Roberts 1994: 78, my italics).

Religion was so present in society that it featured heavily in chymical texts – as well as in other books. However, when the research focus is too narrow, researchers are at risk of overemphasising the importance of things. This is the case of the renowned historian of religions Mircea Eliade (1938; 1962), who managed to contend this myopic view of chrysopoeia in a fashion that seemed unquestionable. He transposed his knowledge of world religions and traditional African metallurgical practices to early chymistry and concluded that, even though the alchemist worked tangentially with chemicals and metals, his real interest concerned the soul: the chemical endeavour of the adept was an attempt at his own transmutation, thereby becoming closer to perfection and to the deity. Only those with the adequate state of body and soul would succeed in the quest and this was, according to Eliade and his followers, the real challenge and the supreme goal.

It should be emphasised that Renaissance written sources do not prove this attractive story. In chymical writings, we do find some statements such as the following:

> "our intention is not directed towards teaching anyone how to make gold but towards something much higher, namely how Nature may be seen and recognized as coming from God, and God in Nature." (Georg von Welling (1655-1727), as cited in Eliade 1962: 165-166).

This, removed from its historical context and ignoring the bulk of the literature surrounding it, might serve to support that the goals of transmutation were beyond this world. However, once contextualised, it looses exceptionality. For a Renaissance person, chymistry and religion were related, insofar as religion impregnated absolutely

everything. Furthermore, investigating nature led to a better appreciation of what was seen as God's wise and generous creation, and hence chymistry – not only chrysopoeia – could be seen as a very respectable occupation. It is also true that it was convenient to have God's favour when embarking on transmutation experiments – or on any other task, at any time! Recent studies across the whole spectrum of the history of science show how the presence and importance of theological concerns were shared by all branches of early modern natural philosophy. As asserted by Debus (1978: 13-14), "it is important not to separate the 'mystical' and the 'scientific'" when studying the natural sciences of this time, since "to do so would be to distort the intellectual climate of the period". This applies to all spheres of activity – the case of chrysopoeia not being special. In sum, a contextual approach to the religious engagement of transmutation shows that far-fetched interpretations are unnecessary. Much simpler and conservative arguments are, on the contrary, supported by the historical record.

c) 'Alchemy' is the projection of imagery from the collective subconscious

Metaphors, emblems and symbols, almost omnipresent in alchemical transmutation sources, were used by Carl Jung as the starting point of his studies. Combining his readings with his experiences as a psychoanalyst, he concluded that "we are called upon to deal, not with chemical experimentations as such, but with something resembling psychic processes expressed in pseudo-chemical language" (Jung 1939: 210; see also Jung 1980). According to Jung, the alchemist's "experience had nothing to do with matter in itself" (Jung 1939: 213), and hence there was no point in trying to decode their texts from a materialistic point of view. In his view, gold makers did not use obscure language to hide actual chemical substances and make the task of the uninitiated difficult; they used the bizarre terminology of dragons, dying kings and strange figures simply because these were the forms in which the collective subconscious erupted into the chymist's mind.

Jung's depiction is, together with Eliade's, the most influential in the common perception of transmutational chymistry, and we find his ideas respected and repeated to different extents in most later studies on the history of science. This approach seems inconsistent, though. Increasingly, more and more of the written sources and emblems analysed by Jung are decoded in modern chemical terms, and the processes even replicated experimentally, without the need of analytical psychology (Read 1936; Obrist 1982; Principe 1987; 2000). Thus "the 'otherness' of alchemical texts rests, then, more

on their modes of expression than on their modes of laboratory work" (Principe 2000: 71). Moreover, there is now evidence of chrysopoeians who used the peculiar allegorical descriptions in their books while being perfectly capable of expressing the very same experiments using clear, explicit, 'chemical' terms in private communications such as letters. All these arguments render the Jungian view very questionable (see also Rodríguez Guerrero 2001; Principe and Newman 2001: 405-406).

Among various possible examples attesting the real 'chemistry' hidden behind 'alchemical' psychology, Basilius Valentinus' *prima clavis* is brought up here. Valentinus is known as a Benedictine monk and adept of the 15th century – even though his books did not emerge until the end of the 16th century – and is regarded as one of the fathers of 'alchemy'. In the first of his *Twelve Keys*, or twelve stages to produce the philosopher's stone, we read:

> "Let the diadem of the king be of pure gold, and let the queen that is united to him in wedlock be chaste and immaculate. If you would operate by means of our bodies, take a fierce grey wolf, which (...) is found in the valleys and mountains of the world, where he roams about savage with hunger. Cast to him the body of the king, and when he has devoured it, burn him entirely to ashes in a great fire. By this process the king will be liberated. And when it has been performed thrice the lion has overcome the wolf, and will find nothing more to devour in him. Thus our body has been rendered fit for the first stage of our work."[3]

At first, the text might sound bizarre and senseless. In a closer psychoanalytical approach, one might argue that it recalls the image of a woman willing to get rid of a dominant man by some violent means, hence possibly relating to the universal subconscious. However, after considering different options, it was possible to replace the symbols by their real referents and decode the paragraph as follows:

> Let the diadem of the king be of pure gold, and let the silver that is alloyed to it be refined. If you would operate by means of our elements, take stibnite, which is found in the valleys and mountains of the world, and is very aggressive. Cast to it the gold, and when it has consumed it, burn the mixture entirely to ashes in a great fire. By this process the gold will be liberated. And when it has been

[3] The "Twelve Keys" appear to have first been published in *Ein kurtz summarischer Tractat, von dem grossen Stein der Uralten...* (Eisleben, 1599). The translation quoted here is taken from Adam McLean's *Alchemy Website* <http://www.levity.com/alchemy/twelvkey.html> [accessed 14.02.2002].

performed thrice, the gold has overcome the stibnite, and [this] will find nothing more to devour in the gold. Thus the gold has been rendered pure for the first stage of our work.

As it reads now, the text is a rather straightforward description of the stibnite process of gold and silver parting. Gold normally contains some traces of silver. If one mixes this impure gold with stibnite (also called antimonite – antimony trisulphide, Sb_2S_3) and the mixture is heated up, the silver is sulphurised and thus separated from the nobler gold, which settles by gravity as a *regulus* under the argentiferous antimonite. This process is documented by 16th-century archaeological remains (Rehren 1996a) and described in several contemporary written sources, for example Lazarus Ercker (Sisco and Smith 1951: 195-198) who, like Valentinus, advises to, after the first run, "remelt the regulus once or twice with fresh antimony". As noted in the previous chapter, the philosopher's stone was understood by many – namely the *spagyric* chymists – as the purest essence of the prime matter, and the adepts were obsessed about the purity of the elements treated. For example, the chymist and monk John Sawtre – citing Pythagoras as his source – writes that a body "cannot be truly transmuted unlesse (*sic.*) that it be reduced into his first matter." (Sawtre, in Afonso V King of Portugal 1651: 27-28). The various metals known to the chymists were not believed to be fundamentally different from each other. Accordingly, the philosopher's stone could be generated from the decomposition and purification of any of them. However, since gold was thought to be the purest metal, and as their concern was extracting the purest part of matter, it was clever to start with gold. Eirenaeus Philalethes (1627-1665) – nowadays identified beyond question as George Starkey (Newman 1994; Newman and Principe 2002) –, will state it clearly in 1655:

"The Stone you seek, wee said, and still affirm,
Is only Gold, brought to so high perfection
As it is possible, which though a firm
Compacted body, yet by Arts direction,
And Natures operation, it is made
A tinging Spirit, which will never fade." (Philalethes 1654/5, pt. 2, bk. 1: 2)

And what better way of obtaining pure metals than refining them by chemical processes? This was, in fact, understood by the copyist who, already in the 17th century,

added some illustrative emblems to Basilius Valentinus' keys and provided even more clues as to how to refine argentiferous gold and decipher the text (fig. 10).

This and other cases exemplify that, by and large, the chymists' images are not upsurges of a collective subconscious, but meaningful metaphors originating from actual observations of real experiments, and encoded due to the reasons explained above. Again, we do not need to resort to human's subconscious to explain this allegoric language.

Figure 10. Emblem accompanying Basilius Valentinus' *prima clavis*. In the middle, the king and the queen (gold and silver), the latter holding three flowers (perhaps the three times that the process has to be repeated). In the bottom-left corner, a wolf (stibnite) above a triangular crucible which could well be used for refining gold with stibnite. On the right, the copyist advanced another process not mentioned by Valentinus: Saturnus (lead) with a cupel where silver is refined, possibly from the stibnite ashes. The artefacts shown under the emblem are a crucible and a bone ash cupel from Oberstockstall (emblem reproduced with kind permission of the Edgar Fahs Smith Collection, University of Pennsylvania Library).

d) Gold makers believed that matter was alive

Finally, another assumption in the interpretation of chrysopoeia that may be questioned is the vitalism or hylozoism. It is common to think that early modern chymists – and often metallurgists – believed that matter was alive, and that this view only came into question in the 17th-century 'Scientific Revolution'. This notion would be one important basis of transmutation. As minerals and metals seemed to grow underground, and also to change when subjected to particular circumstances, it was concluded that matter had life and contained the seeds of its own development. Accordingly, the adept's goal was 'healing' the metals, 'accelerating' their lives so that they would become nobler. This concept, indeed present in some chymical sources, was overly stated by Mircea Eliade after documenting similar beliefs in traditional African metallurgists, and was then followed by many scholars.

The dichotomy between alchemy as vitalist and chemistry as mechanist can no longer be sustained. Principe and Newman have identified practitioners of transmutation stating explicitly that the laws governing the animate world are different from those governing the inanimate world of minerals and metals. In addition, they presented sources whose authors clearly acknowledge that when referring to the 'anima' or 'semen' of the metals, or when discussing male and female principles inherent to different substances, they are simply using metaphors (Principe and Newman 2001). These were allegories utilised to explain their practices within the secrecy of their texts, but cannot be taken as plain and open affirmations. It may be worthwhile to remember that in the present-day world we often explain chemistry in terms of 'excited' and 'attracted' electrons, and this does not mean that we believe that atoms are alive. Renaissance scholars had the very common habit of arguing by means of analogy (Crosland 1962), so these 'humanising' metaphors are even more understandable in their historical context.

This is not to say that these sorts of ideas were completely absent in early modern perceptions of the mineral world. The English alchemist Thomas Norton, writing in the late 15th century, acknowledges an existing debate between those who believe that metals grow and multiply, and those contesting this view (Norton 1652: 18-21). Georgius Agricola does believe that metals develop over time, but he makes the point that this is an asexual, inorganic process, and in this he differentiates himself from earlier alchemists (cf. Halleux 1974). Some Renaissance chymists thus believed that

matter did grow and upgrade over time, and such statements appear in the texts of the time. However, it cannot be assumed that they all did so, since there is evidence of authors seeking metallic transmutation but supporting conflicting views.

2.3. The overlap between chymistry and metallurgy: fire assay

Up to this point, some popular connotations normally attached to the concept of 'alchemy' have been outlined. It has been shown that each of these concepts can be strongly questioned – most even invalidated. We should talk about 'chymists', some of them attempting chrysopoeia, and some not. The quest for the philosopher's stone is but one endeavour – perhaps a particularly interesting one – within the chymical domain. Returning to the particular case study of Oberstockstall, this meant that the interpretation of the archaeological remains should necessarily be conservative. The mere evidence of the practice of chymistry would in itself allow little inference as to the perception of materials, religious sensitivity, psychology or understanding of the world of the artisans involved, irrespective of whether or not transmutation was attempted there. Complementary research strategies would need to be devised in order to investigate this issue. In addition, there was another point yet to be clarified: together with the specialised chymical equipment, the assemblage showed clear evidence of metallurgical fire assays being conducted in the laboratory. How could this combination of activities be explained? What was the relationship between Renaissance chymistry and metallurgy?

At a first glance, one could assume that metallurgy and chymistry had little in common – an idea that underlies most secondary literature. Metallurgy was an industry and metallurgists mere craftsmen, whereas the chymist is seen as a scientist or philosopher involved in a completely different pursuit. Furthermore, metallurgy and chymistry had very different goals. For the chymist, the main enterprise was the examination and understanding of the natural world, and possibly the transmutation of metals, whereas the metallurgist only aimed at the practical exploitation of the mineral wealth. This, combined with the present-day segregation of the two disciplines, may be the reason why early modern chymistry and metallurgy are normally studied by different scholars using different strategies.

What happens when the two domains are looked at together? Obviously, the reactions undertaken by metallurgists are of a chemical nature, and in cases such as

Oberstockstall, as well as in other archaeological and written sources (discussed below), it is clear that chymists often conducted metallurgical operations. The chemical character of early modern metallurgical practices has often been noted (e.g. Stillman 1924; Hoover and Hoover 1950; Sisco and Smith 1951; Leicester 1956; Lange 1965; Smith and Forbes 1969; Rocke 1985; Halleux 1986; Smith and Gnudi 1990; Long 1991; Roberts 1991; Beretta 1993; Multhauf 1993; Rehren 1996a; Rehren 1997; Beretta 1997; Levere 2001; Henry 2002; Rehren and Eckstein 2002; Newman and Principe 2002; Rehren 2002a), nonetheless the actual relationship between both realms, specifically in the field of analytical chemistry, is yet to be explored in detail. When the case of fire assay is considered, this link appears obvious. Early and very significant workshops combining chemical and metallurgical expertise were always found in the official mints, where both analytical chemistry and large-scale metallurgical operations were carried out. This began with the very earliest coinage, introduced in the 7th century BC in Sardis, which within a few decades led to the first development of parting gold from silver (Ramage and Craddock 2000) – a hitherto unknown technology – and certainly did not end in the mid 19th century AD laboratories of the Royal Mint in St Petersburg, where the refining and processing of platinum metal was developed to an industrial scale (Auer *et al.* 1998). The practice of fire assay, however, also extends to less official minting operations (Jeanjacquot 1993); the need for reliable and repeatable analyses, typically by fire assay, is common to all of them. This is not only true for the development of new techniques, but also for its day-to-day practical side. A suitable example for our purpose here is the infamous Frobisher Expedition, almost to the year contemporary to the activities at Oberstockstall. Here, experienced assayers were included in the small fleet of vessels repeatedly sent by Queen Elizabeth I to northeast Canada in search for gold. Based on their analyses, both in London and then in Canada, considerable amounts of worthless rock were mined and shipped back to England for processing; the dispute is unresolved to this day whether the unrealistically high gold assays in the initial samples were fraudulent or due to sheer incompetence (Unglik 2000; Beaudoin and Auger 2004). What is important for us here is the close relationship between chymistry and metallurgy which again becomes apparent.

Assay in general refers to the quantitative determination of the metals present in ores or in metallurgical products. In practice, this comprises a carefully controlled sequence of operations by which one can ascertain what metals exist in a given sample, and in what proportions. Since early times there have been various relatively simple and

reliable assaying methods, such as the touchstone, used to determine the fineness of gold in a given alloy sample by rubbing this on a dark, fine-grained stone and comparing the streak left to those of a set of standards. However, the most reliable and versatile subset of practices is the high-temperature or fire assay. Roughly, the fire assay consists of a small scale reproduction of what could be done in a larger metallurgical operation: the samples can be roasted, concentrated, smelted, melted, fluxed, refined...; the only peculiarity is that here the different raw materials and products are carefully weighed at different stages in order to work out the proportions among them – and also, mainly since later medieval times, the possibility of using better fluxes and strong mineral acids that could not be afforded in the larger scale counterpart.

By recording the behaviour of the sample under controlled circumstances, the assayer can draw conclusions which are relevant to the whole. Therefore, an important practical use of the assay was the assessment of the viability of the large-scale exploitation of an ore deposit, depending on the richness and behaviour of the ore as shown in the analysed sample. This is the application most often studied in historical accounts and identified in archaeological remains, which is not surprising if one recalls Ercker's statement that over two hundred assays were performed every week in the mines of Kuttenberg alone (Sisco and Smith 1951: 43). This application of fire assay becomes crucial, for instance, for the change from the medieval exploitation of small but rich ore deposits scattered within the geological systems, which were then processed separately for each mine, to the early modern exploitation of larger deposits poor in silver, where the detailed assessment of ore quality not only had to guide the mining process, but also the price paid for the ore when delivered to a central smelter for communal processing (cf. Bartels 1997).

The profession of assayer existed as such, and often the term appears reserved for specialists of metallurgical analysis working in mining affairs or coin mints. In the Porto Mint (Portugal), for example, 14th- to 16th-century written documents attest the existence of such specialised and distinct jobs as assayers, counter-assayers, gold refiners and masters of the scale, amongst others (*ensaiadores*, *contra-ensaiadores*, *afinadores de ouro* and *mestres da balança*; (Lopes *et al.* 2000: 59)), the same as known in 13th-century Venice (Stahl 1988; for studies on the organisation of historical mints, see Mayhew and Spufford 1988). However, assay was a much wider concept and – perhaps with the exceptions above – the boundaries among small-scale pyrotechnological specialities were not clear-cut. In the late 17th century, Sir John Pettus (1685, II: 2) notes in his

Essays Explaining the Metallick Words that the same person "is commonly call'd alchimist or chimist, melter, prover, assayer or extracter of quintessences; and such an one was our Author [=Lazarus Ercker!]".

This is possibly due to the fact that fire assay could be of use in many other ways. For instance, as noted before, it could be used for determining the purity of metals in jewellery. In addition, since the noblest part of the sample was extracted, the process could also be seen as a tentative method of purifying matter and extracting quintessences, as seen above in the *first key* to make the philosopher's stone. Fire assay also offered an irrefutable test for the quality of the alleged gold 'alchemically' produced. It was a reliable analytical tool, useful even if only used for investigating the composition of natural minerals for the sake of knowledge. Finally, perhaps some aspiring adepts could perform assays in the belief that they were creating – not just extracting – gold.

There is abundant historical evidence to prove that chymists performed fire assays routinely, even before the use of these methods was so profusely attested in metallurgical workshops. In fact, an account of the written sources reporting the use of sophisticated analytical tests may start in the 10th century – if not before – if alchemical sources are considered (Halleux 1986; Szabadváry 1992; Newman 2000; Newman and Principe 2002: 35-49)[4]. For example, the 13th-century alchemist Paul of Taranto presented a detailed description of the method of cupellation of silver with the aid of a blowpipe that we find practically unchanged in the writings of 16th-century metallurgists:

> "Let a very well sieved cinder be taken and mixed with water of salt; let a vessel be made from it, in which silver or whatever metal that you seek to test in the cupel be put on a very violent fire. With the metal fused, let a sixth part of lead be thrown on; this is especially done in the case of silver. Let a pipe of iron or reed be had, through which one can blow on the surface of the fused metal. The lead fused on the metal will be seen smoking due to this – that it has volatile flight as well as the loss of its substance owing to its badly fixed principles. Thence it is that, passing into smoke, it will draw with it all that is imperfect in the metal to be purged. The purged metal with the lead added to it will be recognized not to be vaporized, but it will seem to be boiling, and to eject froth

[4] These publications include references to early chymical texts covering fire assay practices, which could be useful when interpreting archaeological remains.

– as it were flying froth; then let no more lead be added."(trans. Newman 2000: 36).

Not only were fire assays carried out habitually in Renaissance Europe, these practices would have a crucial role in the development of modern chemistry (Szabadváry and Robinson 1980; Ihde 1984: 277; Szabadváry 1992). Being a small scale test with a vital economic repercussion in its potential large-scale counterpart, the assay demanded maximised efforts in order to ensure precision and accuracy, hence the wide range of tools and methodologies developed to this end and shared by chymists and metallurgists, which would remain fundamentally unchanged until the 20th century (Halleux 1986; Rehren 1996a; Rehren 2001) (figs. 11-16). In addition, inherent to the fire assay is the need for reproducibility. Reproducibility is a key concept of experimentation; and here we can see the seed of modern analytical chemistry (Knight 1992; Rehren 1996a; Principe 2000; Newman 2000). Furthermore, the assay required and enabled the knowledge of chemical substances, their replacement and precipitation reactions under given circumstances and their reproducible nature. Even if it was not formulated explicitly, in practice the assay hinted at the idea of conservation of mass and constant combining proportions (Sisco and Smith 1951: xv). This holds true for both metallurgy and chymistry.

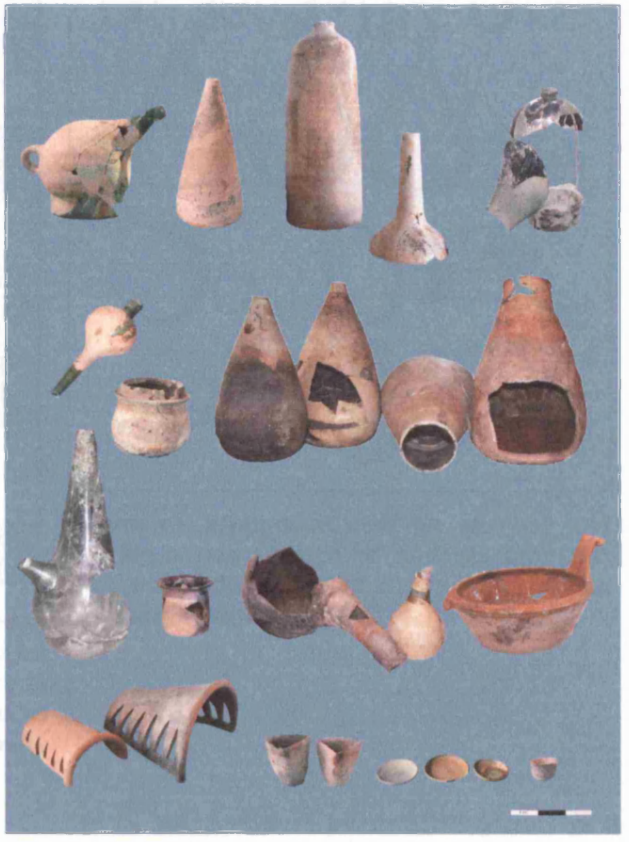


Figure 11. Selection of artefacts from the 16th-century laboratory excavated in Oberstockstall. Scalebar in the bottom-right corner represents approx. 15 cm.

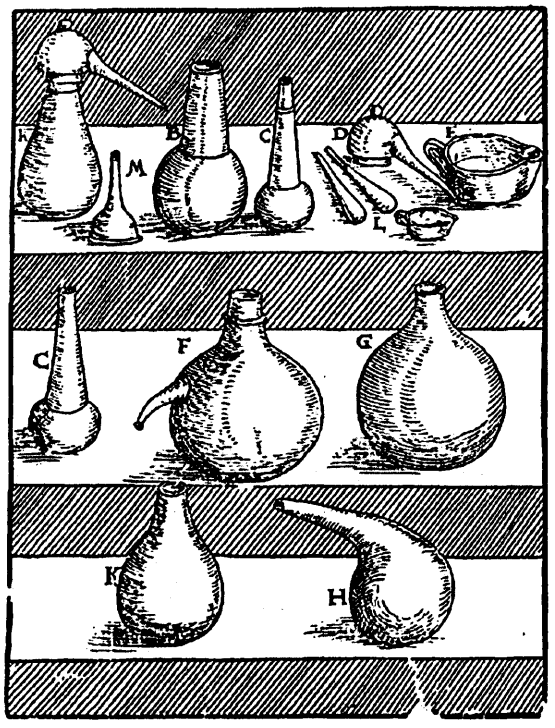


Figure 12. Some glassware and pottery used in assaying and parting, according to the assayer Lazarus Ercker (1580, plate 20). Compare to fig. 11 (reproduced with kind permission of the Edgar Fahs Smith Collection, University of Pennsylvania Library).

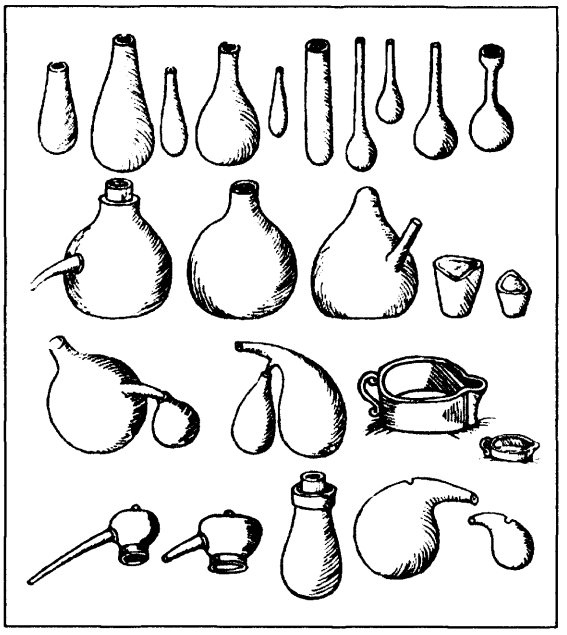


Figure 13. Equipment used for assaying and parting as represented in Andreas Libavius' *Alchemia* (1597, adapted after several plates). Compare to figs. 11 and 12.

While mostly concerned with the practical applications of their activities, mineral assayers approached the notion of 'pure bodies', the precursor of the modern pure elements (Halleux 1986: 281). Moreover, early modern metallurgists discovered new metals (zinc, cobalt, bismuth), which would challenge the traditional chymical conception of *the* seven planets interacting with *the* seven metals (Leicester 1956: 94). Likewise, as the experimentation with minerals developed, it necessarily involved a contradiction of the Aristotelian view of the material world as uniform matter manifested in four simple elements (Halleux 1974; Roberts 1991: 150).

According to the traditional view (e.g. Needham 1974: 20), it might be argued that chymists and metallurgists followed somewhat parallel paths, that some of their techniques and processes happened to be similar but they never looked at each other's work – and thus nor at each other's discoveries. Can we assume that there was such little interaction? Scientists like Philip Melanchthon (1497-1560, as cited in Wightman 1962: 48) investigated "the order, qualities and motions of all bodies and kinds of beings in nature, and the causes of the generation and decay and other motions in the elements

and in other bodies which arise from the intermingling of the elements"; whereas metallurgists such as Vannoccio Biringuccio (1480-ca.1538, in Smith and Gnudi 1990: 87) "avoid multiplying such considerations and disputes (...) all the more because my intention is only to tell you how minerals are found and, once found, how they are extracted from the earth". As Roberts (1991: 151) phrased it, "[the metallurgists'] concerns were thus distinct from those of the explanatory aims of the humanists", and hence we might conclude that both fields were essentially not interested in each other.

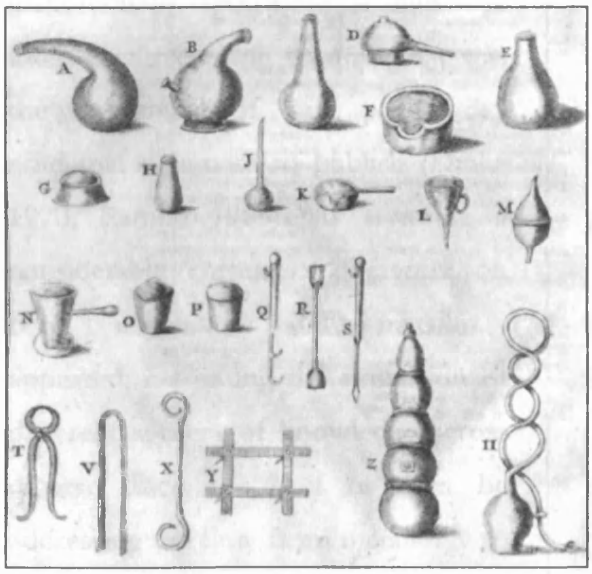


Figure 14. Assaying equipment from Johan Conrad Barchusen's *Pyrosophia* (1698, plate 5). The basic tools remain the same as in early modern times (reproduced with kind permission of the Edgar Fahs Smith Collection, University of Pennsylvania Library).

The picture is not so simple, though. In fact it becomes more complex as cross-disciplinary research develops. The historiographic school of the 'scholar and craftsman thesis' has advocated that the transfer of skills and knowledge among different social spheres played an important role in the development of the experimental method and the natural sciences in general (cf. e.g. Wightman 1950; Wolf 1950; Wightman 1962; Rossi 1970; Wightman 1972; Hall 1983; Henry 1990; Pumfrey *et al.* 1991; Hall 1994; Henry 2002). For example, this happened with the incorporation of discoveries from the military in other social arenas, but also when craftsmen's techniques were adopted by scientists. Indeed, humanist pedagogues such as Luis Vives (1492-1540) already advocated the study of trade and crafts secrets (Henry 2002: 34). However, the input of the 'craft' metallurgy in the 'science' chymistry has hardly been explored from this useful perspective[5]. If one embarks on this task, soon we realise that Renaissance chymists learned from metallurgical assayers, and *vice versa*, meaning that the 'science' could have influenced the 'craft' as well.

[5] Halleux (1986) was an early precursor of this approach, studying the interaction between metallurgy and chymistry. Relevant information regarding this link in the 17th century may be found in the work by Newman and Principe (2002).

The 16th century saw an expansion of printing technology coinciding with many technological and scientific achievements, as well as an emphasis on the practical applications of knowledge; this was accompanied by a vehement – often institutionally sponsored – humanist interest in recording wisdom that was the beginning of our present-day academic eagerness to publish (Drake 1970; Eamon 1985). As a result, a considerable corpus of literature on both chymistry and metallurgy appeared, facilitating the expansion of different sources of knowledge across diverse disciplines. Let us start by addressing the flow from metallurgy to chymistry. As Pamela Long (1991; 2001) has argued, most 16th-century metallurgical treatises were aimed at laymen, in order to defend the status of the mining profession and encourage potential investors. In this sense, they contributed significantly to the "openness of knowledge". Therefore, it is likely that Renaissance chymists read and learned from metallurgical handbooks, which were normally much clearer than the obscure writings by transmutation seekers. Again the case of Georgius Agricola is illustrative, as an established humanist who was deeply interested in mining and metallurgy and wrote in Latin to ensure a wide and learned audience (Hannaway 1992; Beretta 1997). Much of his erudite discussion would be more readily understandable for an educated humanist than for an illiterate craftsman. If we bear in mind that during the Renaissance humanism and science cannot be separated (cf. e.g. Blair and Grafton 1992), it follows that Agricola's observations may well have fed scientific thoughts. Indeed, his works are to be found in the libraries of the most

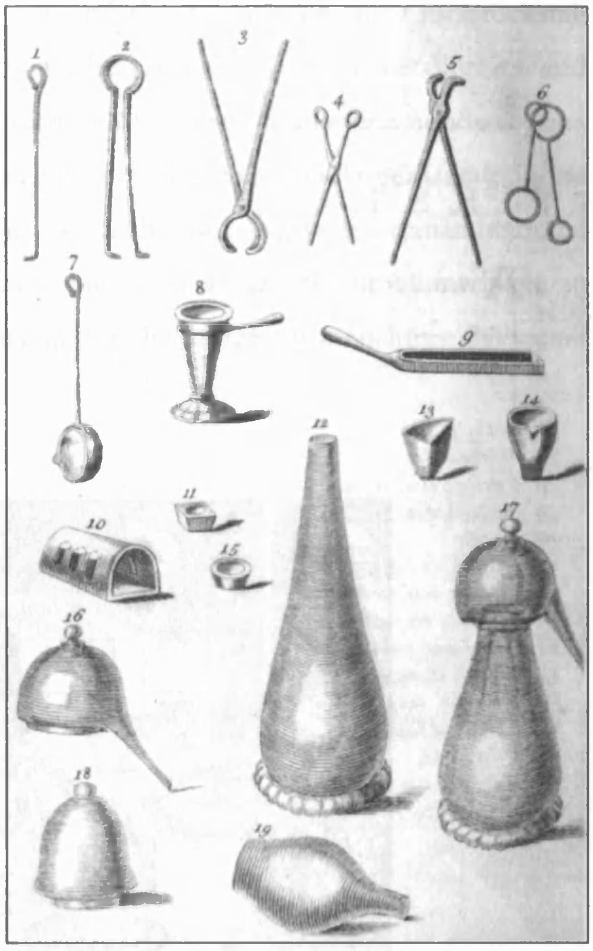


Figure 15. Assaying equipment depicted in William Lewis' *Course of practical chemistry* (1746, plate 1). Almost two hundred years after Ercker's treatise, the set of assaying tools used by metallurgists and chymists remains basically unchanged (reproduced with kind permission of the Edgar Fahs Smith Collection, University of Pennsylvania Library).

pre-eminent chymists (Beretta 1997). Significantly, a book inventory conducted in 1561 in the library of Urban von Trenbach, one of the patrons of the Oberstockstall laboratory, attests the presence of Agricola's publications among other metallurgical and chymical works (pers. comm. S. von Osten 2003). Moreover, almost one hundred years later, when one would expect different disciplines to be more clearly demarcated, one finds George Starkey – who would become one of the most renowned transmutational chymists of his time – spending the money earned as a practitioner of medicine to generously pay a skilled craftsman in exchange for the secrets of metallurgy (Newman and Principe 2002: 160).

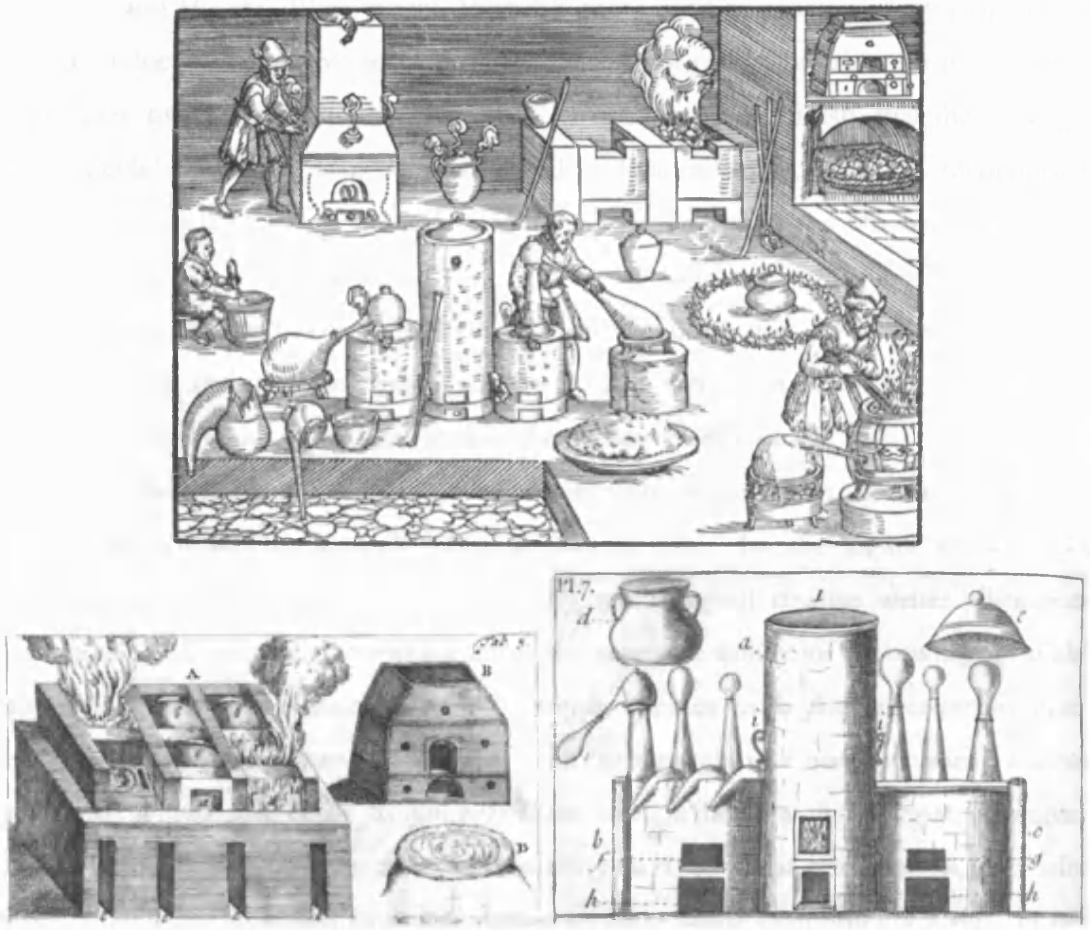


Figure 16. Top, assay laboratory showing different types of furnaces, from Ercker's *Treatise on ores and assaying* (1580, plate 1). Bottom, Different assay furnaces from Barchusen's *Pyrosophia* (1698), still bearing strong resemblances to the ones depicted by Ercker.

This stream from metallurgy to chymistry fostered by the socio-economic atmosphere of the Renaissance must have also occurred in the opposite direction. Even though Agricola is mostly concerned with metallurgical practices, we can infer from his

comments (Hoover and Hoover 1950: xvii ff.) that he read literature of all sorts, including chymistry, and in fact a friend of his reports that he suffered from conjunctivitis due to "untiring study and insatiable reading" (Rocke 1985: 40). His concern with chrysopoeia demonstrates that he was acquainted with the practices of other people who processed minerals and metals to pursue objectives different from his own. It is remarkable that his disapproval of gold makers did not target the unfeasibility of their claims, but rather their obscure and un-etymological language, together with the frauds of those who deliberately tried to cheat, these "warranting capital punishment" (Hoover and Hoover 1950: xxix). As to the gold making attempts themselves, Agricola appears rather unsure: "Whether they can do these things or not I cannot decide" (Hoover and Hoover 1950: xxviii). Probably more used to practical demonstrations, he appears reluctant to believe something that he cannot prove, but the idea itself does not seem alien to his conceptual framework[6]. Let us mention, to finish, that the cover page of Agricola's *De re metallica* is ornamented with a caduceus, which is related to the divining rod and other potent rods traditionally attributed to the chymists (Read 1936: 79).

The connection between chymistry and metallurgy may go further. If we assume that a mineral and metal assayer would be familiar with most of the chemical reactions normally conducted in the quest for the philosopher's stone, and that their ideas regarding the origins and behaviour of metals were similar, then we may wonder: why would he not try for himself? The answer is given by the figure of Vannoccio Biringuccio, another renowned 16th-century metallurgical treatise writer. Biringuccio only started to criticise chrysopoeia after the repeated failure of his own gold-making attempts (Smith and Gnudi 1990: 37), which stresses both the cross-fertilisation in knowledge and techniques and the fact that chrysopoeia was not necessarily a strange quest for a practitioner of metallurgy. Even though he is a metallurgist, Biringuccio discusses extensively his concerns with other practitioners of chrysopoeia, and admits that "I have not refrained from attempting to make some experiments myself in order to understand it better" (Smith and Gnudi 1990: 37). Furthermore, Biringuccio only finds transmutation impossible insofar as it would require conditions, such as perfect

[6] Agricola does criticise some earlier explanations of the nature and formation of metals, and questions the feasibility of transmutation. However, he does not go on to state that transmutation is impossible. Therefore, contrary to other authors (Lange 1965; Halleux 1974), I do not see Agricola as unequivocally opposed to "alchemy" altogether. Some of his positive remarks regarding this discipline are mentioned later in this chapter. This, or course, just underlines that Renaissance chymists did *not* (yet) distinguish between 'bad, impossible' alchemy and 'good, real' chemistry in the way we do today.

crucibles and furnaces and a very constant fire (Smith and Gnudi 1990: 41), impossible for a 16th-century laboratory. This highlights a deep concern over the development of high quality equipment.

Just as Biringuccio represents the metallurgist meddling in chymistry, so does Count Wolfgang II von Hohenlohe (1546-1610) embody the reverse example: aristocrat, chymist and concerned with the secrets of nature, he was however very familiar with the necessary skills and therefore could kindly assay ores for his friends (Weyer 1992). In an opuscule on the philosopher's stone published in 1651, John Sawtre quite clearly advises chymists to look at craftsmen's dexterities:

> "(...) and after that thou hast it [the 'yellow body'] well purged, beat it most strongly, and utterly, and make it into thin plates, and after beat them into leaves, the thinnest that can bee possible, *as gold-beaters doe.*" (Sawtre, in Afonso V King of Portugal 1651: 24, my italics)

Another person who experienced the intersection between both fields was Lazarus Ercker (*ca.* 1530-1594). Assayer by profession, Ercker used his reputation to promote himself until he was appointed by Elector Augustus at Dresden in 1555. Augustus was an enthusiast of both metallurgy and chymistry and maintained a laboratory in his resident castle (Long 1991), thus we may hypothesise that Ercker had the chance to see – perhaps even try – transmutation experiments there. This might be the reason why, when writing his famous treatise on assaying two decades later, he stressed that chrysopoetic issues "have no place in this discussion of cementation, since my books will contain nothing but natural and tested processes which can be relied upon and raise no false hopes" (Sisco and Smith 1951: 190). In any case, Ercker would again have the opportunity to work among chymists when he was appointed by the court of the emperor Rudolf II of Prague, who was renowned for his deep belief in, and patronage of, chrysopoeia. Significantly, while working as courier of mining affairs, the assayer kept his old concerns while exchanging correspondence "on alchemical matters" (Evans 1997: 215).

Finally, it should be mentioned that not only did metallurgists read chymical books and sometimes practise chrysopoeia, but they also learned from chymists' experiments. The use of distillation for making their own parting acids, as described in the foremost assaying treatises, illustrates a technique that had much earlier been designed and utilised by chymists (cf. Forbes 1970). Sometimes, metallurgists directly

acknowledge chymists' achievements. Agricola (1556), for example, mentions *chymistae* as inventors of the method of parting gold and silver by nitric acid. Also Biringuccio (1540) praises *alchimisti* as discoverers of techniques such as brass making or sublimation, and as masters of soldering, enamelling, working in niello, gilding and so forth. Even though these testimonies should not be taken at face value (Halleux 1986), their very presence illustrates once again that the flow of knowledge took place in both directions. Contrary to the commonplace of secondary literature, chymists were taken as a serious source of know-how and practical experience, and even chrysopoeians were not necessarily rejected as a group.

2.4. Summary

The questions originally posed when addressing the archaeological remains of the Renaissance laboratory in Oberstockstall have led to several observations which may be of relevance for a wider spectrum of scholars. Firstly, this discussion has emphasised the important role of fire assay in the period expansion of mining and metallurgy, as well as in the development of modern experimental science. Besides offering various practical applications, the analytical observations enabled by the fire assay would foster a new understanding of the nature of minerals and elements. Accordingly, this repertoire of techniques should be studied not only in the context of the history of metallurgy but also within the history of science in general.

Secondly, this chapter has shown that, especially when dealing with fire assay practices, the boundaries between chymistry and metallurgy seem very permeable. In some instances, both domains are indistinguishable – as they were for Renaissance peoples. We cannot consider either side without resorting to the other, and a historical appraisal of fire assay will only be complete if both metallurgical and chymical sources are equally considered. Otherwise, we will be transposing anachronistic, present-day categories to a biased reconstruction of the past picture. This revised approach provides a more adequate basis for the interpretation of archaeological remains, and *vice versa*.

Thirdly, if we are to embark on this more comprehensive reconstruction of past assaying theories and practices, we should try to avoid traditional presumptions and confusing terminologies that have come into question. The inclusive term 'chymistry' provides a useful alternative for the misleading 'alchemy' and 'chemistry', since only one indistinct discipline existed until the end of the 17th century. The attempts to transmute

base metals into gold ('chrysopoeia') or silver ('argyropoeia') constituted but one activity practised by both chymists and metallurgists. This practice did not bear any more of the magical, religious or psychological connotations than usual for the time, and conversely it did involve the systematic conduction of fire assays. Only towards the end of the Renaissance, the very experiments in the quest for the philosopher's stone would demonstrate that this was an impossible task, thereby paving the way for the future segregation between modern chemists and outdated Aristotelian alchemists.

The transposition of present-day categories in the reconstruction of the past is an omnipresent risk in historical sciences. The vast majority of studies on metallurgy, 'alchemy' and 'chemistry' for the period concerned suffer from the hindrances pointed out above. It appears to the author that the strategy to overcome them is twofold: on the one hand, the boundaries among present-day disciplines must be crossed and the problems tackled from a more holistic perspective; on the other hand, regardless of the subject to be investigated, a contextual approach is advisable, since no aspect of history can be separated from its circumstance. In this way, the resulting image of the past will be more comprehensive and hopefully more accurate.

This doctoral thesis shall not constitute the definitive study of the material remains recovered in Oberstockstall, as surely others will come to complete and correct it in various ways. However, this approach has allowed a clearer definition of some important issues as they regard fire assay and, by combining scientific analysis with a review of literary sources, traditional biases are being avoided. It is hoped that any other approach to this assemblage shall be placed in the methodological, epistemological and historical framework here defined. The author eagerly expects that such an approach will contribute to our understanding of Renaissance chymical practice as much as its underlying theory, and also that it will encourage more historical, archaeological and scientific research along this line.

3. Towards a scientific archaeology of alchemy and chemistry

"All hope for the good and often it turns out well. No one truly with an abject and timid soul ever did anything or indeed ever will do anything."
Georgius Agricola (1494-1555)

3.1. Introduction

In the last decades, contributions from complementary backgrounds have enhanced our knowledge of the history of alchemy and chemistry. What once was largely the pastime of erudite professional chemists has increasingly become the pursuit of social historians, historians of ideas, philosophers, historians of science, technologists or full-time historians of alchemy and chemistry. In the previous chapters, the possibility was suggested of adding yet another professional to this list: the archaeologist. The inclusion of archaeology in the picture already prompted a new focus and led to several observations that are relevant for the entire discipline and had, however, been largely overlooked. In this chapter, the potential of using archaeological materials and approaches to study the history of alchemy and chemistry will be explored in more detail, with a special emphasis on the period and issues concerned here, *i.e.* Renaissance chymistry. After outlining and discussing some problems entailed by traditional approaches, it will be argued how archaeology could help overcome them. The rest of this thesis is presented as a first step towards a practical demonstration.

3.2. Traditional approaches, traditional problems

The main source of information for the history of alchemy and chemistry is a relatively large corpus of treatises, correspondence and other writings, which keeps growing as a result of discoveries in libraries and archives. Their exploitation has proven very fruitful and, undeniably, many studies exclusively based on these sources still

remain as classic. However, a review of the main works in this field allows us to identify several limitations.

A typical bias of text-based studies is the stress on theoretical debates that tend to ignore the practical side of the discipline. To name but two examples, science historians have often focussed on topics such as the role of magic and religion in the development of modern science (e.g. Burtt 1932; Thorndike 1923;Rabb 1965; Rabb 1966; Debus 1978; Vickers 1984; Henry 1990; 2002; Webster 1996), or the dispute between Paracelsians and Aristotelians (e.g. Pagel 1958; Debus 1965; 1978; 1991; Roper 1985; Webster 1996; Grell 1998). Research on such issues provides rich insight into the cultural atmosphere of the time and the wider engagement of the chymical endeavour. Nevertheless, often this emphasis on the theoretical side fails to notice that chymistry was, above all, an *applied science*. Despite the abstract appearance of many writings and their broad social and cultural implications, Renaissance chymists' thoughts mostly originated from laboratory activities, and always bore in mind the practical applications of their knowledge. Thus, our studies should start by investigating these laboratories, including the reactions and observations carried out, which were the real seeds of the chymists' discourses.

In addition, historical studies often concentrate on the biographies of main figures that are recorded in the historical development of chemistry, and use them as a chronological backbone (e.g. Holmyard 1928; Partington 1960; 1962; Farber 1961; Hannaway 1975; Debus 1991; Levere 1994). In this sense, they have made "an increasingly austere discipline more human, providing role models and showing how progress in chemistry was usually dialectical rather than straight-line" (Knight 1992: 3). However, at the same time, many common chymists who might represent the actual state of affairs of the time are overlooked, just because they were not outstanding or because their writings, if existed, have not survived. As in other disciplines, this has resulted in a history of chemistry characterised by a sequence of achievements, more or less separated chronologically, that overlooks the day-to-day, repetitive, trial-and-error nature of scientific development.

A further, subsidiary, source of information has been the corpus of depictions representing transmutation laboratories and symbols, chymical instrumentation or related scenes (e.g. extensive use in Ferchl and Süssenguth 1939; Read 1947; Holmyard 1968; De Rola 1988). The value of this source material is limited too, thus it should be used with caution. Illustrations in treatises may well be later additions to much older

manuscripts (Anderson 1985: 219), they may be expressions of a metaphorical language or respond to specific cultural trends (Obrist 2003). The major aim of the Renaissance painters who depicted chymists was to fulfil customers' expectations, not to represent or document reality. Indeed, an art history approach to these paintings reveals that their nature tended to be primarily allegorical and didactic. This, among other drawbacks (cf. Hill 1975; Principe and De Witt 2002), challenges the reliability of such illustrations for historical reconstructions and stresses the need for complementary approaches.

3.3. Instruments, experiments and laboratories

The scientific instrument materialises the link between theory and experience. This crucial role of the instrument in the functioning and development of modern science has been noticed for several decades. As emphasised by Abraham Wolf (1950: 9),

> "One of the chief characteristics of modern science consists in its use of scientific instruments. The function of such instruments is various. They may enable the observer to observe much better what he can already observe with his unaided senses, though not so well. They may enable him to perceive something that would otherwise be entirely imperceptible. They facilitate the precise measurement of phenomena. Or they may make it possible to study a phenomenon under conditions so controlled as to justify reliable conclusions about it (...). Moreover, the measurement and the quantitative correlation of phenomena play so important a role in modern science that one can hardly imagine the very existence of modern science without the aid of above-mentioned and similar scientific instruments."

What we may consider as an instrument ranges from a magnifying glass to a sophisticated transmitted electron microscope, from a flask to a vacuum chamber – it is its 'instrumental use' that characterises an instrument. Clearly, science cannot function without instruments, and observations such as Wolf's are somewhat acknowledged or repeated in many studies on the history of science in general, and chymistry in particular (Child 1940; van Helden 1983; Anderson 1985; Taylor 1986; Anderson 2000; Principe 2000). Some have broadened the scope to elaborate on the emergence and development of laboratories as the essential workplaces where science was created. In this vein, the

work by Owen Hannaway (1986) is remarkable, as he traced the Renaissance origins of the *laboratorium* as first related exclusively to chymistry, and showed how this enabled the vital step from possessing *scientia* to doing science.

The problem is that this kind of statements tend to appear just as 'cautionary tales': after stressing the important role of the instrument and the laboratory, the historian goes on to tell the history of the discipline as a succession of individuals and conflicting theories. Some scholars have attempted alternative approaches such as writing a history of the laboratory or its equipment (e.g. for chemistry, Holmyard 1956; Needham 1974; Needham 1980; Krätz 1982), or even encouraging and documenting the preservation of old chemical instruments (Anderson 1985; Stock 1986). Although interesting perspectives, these again dissociate laboratory from science: on the one hand, we have the history of the laboratory; on the other hand, the history of science. More recently, an attempt has been made to rectify this imbalance between theory and practice in the historiography of chemistry, under the eloquent title *Instruments and experimentation in the history of chemistry* (Holmes and Levere 2000). The contributions to this volume are commendable in that they contextualise instruments and experiments within their wider historical contexts, and explain their responsibility in certain discoveries and scientific developments.

There is, however, much more potential for these studies. For example, they should pay more attention to the *active* role of instruments and experiments. As shown by Yakob Rabkin (1993: 28),

> "scientific instruments, even when these are produced at the express request of scientists and in accordance with their specifications, do not remain passive tools in the hands of the active researcher. Physics offer abundant examples of how a new measuring device can 'boost' a field of research".

Instruments not only enable the solution of problems as required and designed by researchers: they also *discover*, often unexpectedly, new solutions – or at least new problems – that are the seeds of scientific progress. It suffices to remember here how modern chemistry emerged as an offspring of the instruments and experiments used for transmutation, coin minting or metallurgical analysis. As it will be shown, there are also more 'subtle' ways in which, in a wider sense, artefacts play a central role in society.

Yet there are more lines to develop from the focus on the laboratory, but they will require adequate theoretical tools and research strategies. In the author's view, these

can be learned from two complementary fields: archaeological and anthropological studies of material culture, and sociohistorical studies of technology.

3.4. From *inspiration* to *information*: the contribution of (scientific) archaeology

By focussing on material remains, archaeology opens a window to the main and predominantly overlooked side of chymistry: its practice. This may allow us to verify the actual instruments used and processes conducted in the laboratory, and even to assess their relative success. The potential of archaeological information is, in this sense, only comparable to that of the few laboratory notebooks preserved (cf. Weyer 1992; Newman and Principe 2002).

Besides, a more thorough knowledge of the chymists' particular operations may be the clue to understand their theoretical discussions, as the latter are likely to be rooted in the former. This is particularly promising in what regards the allegorical texts written by transmutation seekers. The material remains of chymical activities, if adequately analysed, may appear a great deal more explicit, objective and unveiled than the writings of the people who generated them. If the traces of use are identified, then we have a limited number of options as to the materials processed. The deciphering of chymical texts and emblems should become easier from this starting point.

Not only may archaeological evidence help understand the information given in traditional sources, but also it may correct or complete this before it is confidently accepted by historians. For example, archaeological remains could confirm whether a piece of equipment depicted in the engraving of a 13th-century copy of a 4th-century manuscript did exist in the 4th century or, conversely, whether an apparatus depicted in a 17th-century genre painting appears there as an aesthetic convention copied from earlier painters, even if no longer in use. If we look at the similarities between the furnaces shown in figure 16 (p. 56), we may wonder: was furnace design so conservative for two hundred years, or is this simply the result of one author copying from another? Archaeology may have the answer to this.

Paradoxally, another aspect of the archaeology of chymistry that may turn out to be advantageous is the arbitrary pattern of preservation and discovery of the archaeological record. One of the chymists working in Oberstockstall, for example, was an economically rather modest, hitherto unknown priest, who left a huge debt with the

local apothecary when he died[7]. The picture of everyday early modern chymistry as obtained from his laboratory equipment will probably be a more realistic representation of his time than that given by an exceptional Paracelsus. Cases such as this may help redress the aggrandisement of some particular figures in the historiography, and we should not forget that, as stated by Knight (1992: 7), "science after all is not just a matter of geniuses in garrets". Archaeology does not focus on the work of those authors of outstanding discoveries, nor on those who wrote more books or whose writings have been preferentially preserved – or discovered. Instead, it allows a closer look into a single fortuitous case study, placing it into a wider context. Accordingly, we may identify individual choices and variations of the 'canon' recorded in books, together with virtual trials and failures. The incompleteness of the archaeological record is of a different nature from the incompleteness of written and iconographic sources. Consequently, diverse approaches can help fill the gaps left by each other and the picture of past chymistry may appear broader and richer.

Another benefit of an archaeological approach to chymistry is that archaeology ignores *a priori* the distinctions between metallurgy and chymistry, and hence researchers necessarily become aware of their intersection. The archaeologist focuses on chemical processes and utensils placed in an archaeological context, regardless of their ascription. Research concentrates on the analysis of the artefacts and debris, the interpretation of which requires a broader and more flexible reference framework, and obliges a look at all possible sources. Archaeological and written sources help interpret each other, and only once we know what the artisan was doing can we relate their activity to the directions given in specific treatises. Consequently, at this stage we may have a more consistent foundation to deduce, for example, whether a given artisan was more concerned with applied metallurgy or pure chymistry. Once the archaeology of chymistry is more established, we might be able to verify the importance of different schools and trends, according to the relative evidence of chymists performing and perfecting specific reactions, some of which may have been promoted by particular writers.

Although many metallurgical processes involve chemical transformations of matter and are as such worth studying from the chymistry history (and archaeology) viewpoint, research on fire assay remains is considered particularly relevant. As

[7] Christoph von Trenbach left debts amounting to 22.000 fl., 4.000 of which were owed to the apothecary Wolfgang Kappler of Krems (cf. von Osten 1998: 92).

explained above, we can expect any particular assayer to have maximised resources and efforts in order to investigate the nature of the materials involved, test their properties, experiment, measure the results and eventually replicate processes. Accordingly, it is likely that many of the feats of the analytical chemistry of the time were achieved in this particular field. This, together with the role of fire assay at the interface between metallurgy and chymistry, make the study of this type of laboratories especially promising.

Finally, archaeology carries a theoretical background and a range of analytical models that could facilitate our reconstruction and interpretation of past alchemy and chemistry. In the next section, some pioneer studies of archaeological remains related to alchemy and chemistry will be outlined. Subsequently, a tentative model shall be proposed, aimed at the integration of archaeological and sociohistorical approaches to this field of study.

3.5. Background to the archaeology of alchemy and chemistry

As far as I am aware, the first author who encouraged the study of remains of past chemical experiments was Berthelot ([1905]1968), just one century ago. More recently, Moorhouse (1972) published a groundbreaking article in which, together with some collaborators, he compiled for the first time several collections of British medieval glass and pottery distilling equipment. This landmark publication discussed technical aspects of the process, reported findings and provided the first analytical results on archaeological remains related to chymistry, in the hope that this would stimulate further research in the field. Also noteworthy are Pittioni's (1975; 1978; 1985) novel studies of fire assay remains.

Distillation – and, to a lesser extent, sublimation – has been the chemical process addressed on more occasions, often with extensive reference to archaeological remains (Moorhouse 1972; Forbes 1970; Needham 1974; 1980; Anderson 1985; Rouaze 1989; Thomas 1992; Soukup and Mayer 1997; von Osten 1998; Kamber *et al.* 1998; Weyer 1998; Anderson 2000), which is understandable insofar as some of the utensils used for these operations have very diagnostic shapes, and are thus more readily identified in archaeological contexts. Among these studies, Thomas' (1992) compilation of archaeological remains of al/chemical equipment may be highlighted, as valuable groundwork that should now be completed with the necessary contextualisation and

analytical study. However, with a few remarkable exceptions (Moorhouse 1972; Rouaze 1989; Soukup and Mayer 1997; Kamber *et al.* 1998), scholars have not undertaken detailed scientific analyses of these early pieces of equipment. This may be related to the inexistence of a tradition of studies in this field and the lack of clear research questions. As a result, these publications normally show the archaeological finds merely as illustrations of what appeared in the written or iconographic sources of the time, but adding little extra information.

In 2000, Robert Anderson published an article expressively entitled "The archaeology of chemistry". As a chemistry historian, Anderson started to realise the informative potential of the material culture of early laboratories. His argument came to complete the observations by Thilo Rehren (1996a; 2001; 2002a; Rehren and Eckstein 2002) who, from the experience of a scientific archaeologist, also noticed that archaeological remains could provide details of early chemical processes and help clarify the relationship between alchemy, chemistry and metallurgy. Neither of these scholars went on to define a research strategy to this end, and both of them were slightly suffused with the traditional assumptions attached to 'alchemy' and questioned above (but see Martinón-Torres and Rehren 2003; 2004a). Nonetheless, their pioneer work is a major inspiration underlying this thesis, and it also prompted the author to try to increase archaeologists' awareness of the importance of chymistry remains, their recovery and identification (Martinón-Torres 2003).

Also relevant to this study is the increasing number of published analyses of archaeological remains of fire assays and refining in general (e.g. Éluère *et al.* 1989; Bayley and Barclay 1990; Bayley *et al.* 1991; Bayley 1991a; 1991b; 1992; Eckstein *et al.* 1994; Sperl 1996; Rehren 1996a; 1998; 2001; Bayley and Eckstein 1997; Kamber *et al.* 1998; Schifer 1998; Rehren and Kraus 1999; Unglik 2000; Ramage and Craddock 2000; Rehren and Eckstein 2002). These habitually concentrate on the identification of metallurgical debris from a given site, but pay little consideration to the wider social implications of the facts identified – hence overlooking most primary and secondary literature on chymistry. However, they have significantly helped understand how the processes of assaying and refining noble metals were actually carried out since Roman times, and started to characterise the set of tools and raw materials utilised. Crucially, these studies have evidenced the potential of, and need for, further research in this area, and the necessity of a more comprehensive and systematic approach.

3.6. How to proceed? Theoretical and epistemological framework

There appears to be a myriad of ways in which archaeology, both as inspiration and as information, may help overcome traditional historiographic problems. However, notwithstanding the advances in this direction noted above, these contributions appear as fragmentary pieces in a vast puzzle; the various approaches lack coherent integration within a research programme. In my view, this amalgamation will require sharing analytical categories and theoretical models.

The forgoing review of the current state of affairs has already revealed some gaps left by previous research, and suggested strategies to fill them. Namely, the *craftsman and scholar thesis* has proved a useful notion, especially when we take into account not only the flow from technology to science, but also the opposite direction. From this standpoint, *chymistry* has been defined as an entirety that includes both 'alchemy' and 'chemistry'; its close relationship with metallurgy has been clarified, and assumptions traditionally attached to chrysopoeia or transmutation have been contested.

Furthermore, some concepts from *anthropological and archaeological studies of material culture* may be useful in this field. I expect this thesis to be placed in the general framework of *chaîne opératoire* studies (see Martinón-Torres 2002 and references therein). However, reconstructing full technological sequences, from the provision of raw materials to the end deposition of discarded artefacts, is not the major aim of this thesis. Rather, when looking at fire assay practices, this work shall be considering assay as a *sequence* that directly engages materials, tools, energy, techniques, ideas and gestures. This is what makes the ideal concept of the *chaîne opératoire* different from a bare technical chain or succession of actions. Thinking in terms of the *chaîne opératoire* involves giving equal consideration to all the factors involved: not only what the agent wants to achieve, but also the materials utilised and their interaction, technical knowledge and skills. More importantly, as argued elsewhere (Martinón-Torres 2002), this *chaîne* is not only 'long' – as a sequence of stages –, but it is also 'wide', as it incorporates issues such as agency, innovation, relativism and cultural implications, thus placing past technologies in their wider contexts (fig. 17). Briefly, this means that all the elements integrated in the *chaîne* are conditioned by their circumstances.

A hypothetic example may illustrate some of these concepts: we could consider a given chymist as an agent – ultimately, he is the one that undertakes to produce *aqua fortis* (nitric acid) by distilling a mixture of vitriol (iron sulphate), saltpetre (sodium

nitrate) and water. Possibly, he wants to do it because he has read an allegorical recipe that, for some contingent reasons, ended up in his library, and he deciphered the distillation process in that particular way. On the whole, he believes, this is one step in the production of the philosopher's stone, that enigmatic substance fancied by many people – including himself. He needs an alembic and, perhaps, he will not use the one most resistant to fire, but simply the only one that his local glassmaker could obtain for him in the regional market. In the end, this apparatus will allow the production of a strong acid; indeed, the saltpetre that he bought while peregrinating to Santiago de Compostela was very pure and has yielded excellent *aqua fortis*. The chymist will not achieve metallic transmutation but, in turn, he will find out that this acid is useful for separating gold from silver. Disappointed, he will abandon chrysopoeia, but soon he will find a job as an assayer at a nearby mint recently set up by the emperor. In the future, a Venetian itinerant glassmaker will offer him a much better, newly designed, still head. Doubtful, he will decline the offer: the one that he has, works.

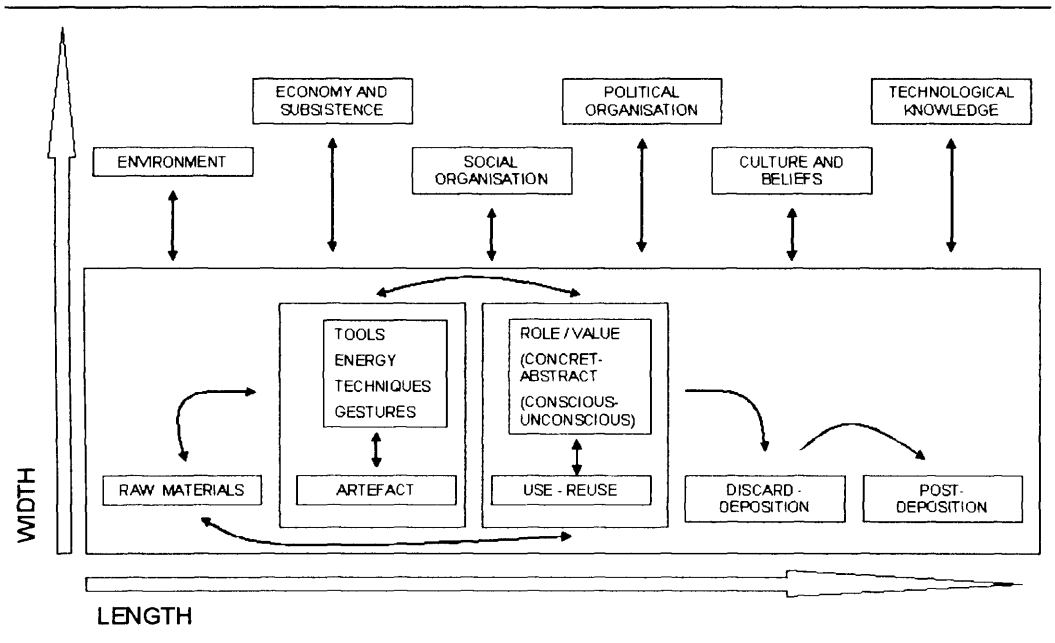


Figure 17. Diagrammatic representation of the 'length' and the 'width' of the chaîne opératoire (from Martinón-Torres 2002; modified and completed after Sillar and Tite 2000: 6, fig. 1; Grace, n.d.).

Admittedly, this story forces too many eventualities in one single anecdote. Nonetheless, it does not lack verosimility and, in it, we find some of the elements that appear inextricably linked in the *chaîne*. Just as much as the individual and his know-how,

raw materials and instruments are fundamental. Materials not only enable planned processes – in this case, a step towards the philosopher's stone – but also affect the course of actions and their outcomes – for example, to discover *aqua fortis* as a parting acid and, ultimately, to change the life-history of the chymist. Overall, social, economic, cultural and other factors, form a particular context that affects – and is affected by – those elements constituting the *chaîne*. This story develops in a certain way because the emperor set up a mint for particular reasons, and the itinerant glassmaker offers still heads because they are demanded at this specific time. We could even discuss why the glassmaker is Venetian. Innumerable contingencies are intertwined. It is valid to deconstruct this *chaîne* and focus on an individual aspect such as the manufacture of alembics or the fineness of Spanish saltpetre. However, ideally, any study should be integrated within this analytical framework, and interpreted in its context.

Another crucial concept exemplified by this story is that of *technological choice* (Shanks and Tilley 1987; Schiffer and Skibo 1987; van der Leeuw 1993; Lemonnier 1993; Schiffer and Skibo 1997; Sillar and Tite 2000; Schiffer *et al.* 2001; Skibo and Schiffer 2001)[8]. Despite the apparent practical orientation of technology and science, there are alternative ways of achieving similar results and it should not be assumed that the easiest, or the economically most profitable, is the one always chosen. This applies to the manufacturers as much as to the users of any item. Technological choices are made at every stage of any given *chaîne opératoire*, and many factors may be conditioning them, from availability and knowledge to cultural and religious values. In the story above, for instance, custom is the main factor leading the chymist to keep his old alembic and refuse the new one. Several scholars have studied technological choices within different spheres of activity, from traps, weapons, house enclosures and clothes (Lemonnier 1986), through irrigation settlements (Pfaffenberger 1988) and up to pottery production (e.g. van der Leeuw 1984; 1993; Cobas Fernández and Prieto Martínez 1998; Prieto Martínez 1999; Livingstone Smith 2000; Pool 2000; Sillar 2000a; Tite *et al.* 2001; Tite and Kilikoglou 2002) or lithic technology (e.g. Perlès 1987; Sellet 1993; Edmonds 1995; Grace n.d.).

Sillar and Tite (2000) have persuasively argued the "challenge" that this notion poses for materials science approaches to past technologies. As far as possible, and

[8] Although Michael Schiffer and co-workers prefer the terms 'behavioral chain' and 'technical choice', sligthly different in their connotations from '*chaîne opératoire*' and 'technological choice', both perspectives are treated here inclusively, as a theoretical approach concerned with the study and interpretation of archaeological material culture and technology within society.

notwithstanding the lack of a standard method to do this (cf. Roux 2001), this thesis shall strive to face the challenge, identify choices and explore the reasons behind them. In so doing, it will be crucial to bear in mind three different analytical categories (cf. Schiffer and Skibo 1997; Sillar and Tite 2000; Skibo and Schiffer 2001; Sillar 2003; Schiffer 2003): formal properties, material properties and performance characteristics, all three intertwined with technological choices. Drawing again from the hypothetical example above, we may start our study by characterising the shape and thickness of the still head – *i.e.* some of its formal properties. These and other measurements will inform us about the material properties of the instrument, such as capacity, thermal refractoriness or resistance to corrosion. We will then need to investigate the specific conditions of use of the still head in order to assess its performance, and to find out which specific formal and material properties were really at stake. For instance, if the still head was used for distilling whisky, corrosion resistance would not be as important a factor as if the product is nitric acid. Finally, it will be necessary to compare this instrument to other artefacts in and outside this laboratory, and to explore the wider context, in order to explore to what extent the chymist was aware of (or concerned with) these different properties, and to discern the possible reasons behind choices in the manufacture, acquisition and use of the instruments. The intricacies of these concepts, as well as the limitations of 'multipurpose explanatory tools' such as function vs. style, will become clearer in the presentation and discussion of the analytical data.

Interestingly, the concepts of *chaîne opératoire* and *technological choice* suitably overlap with other ideas from *sociohistorical technology studies*. The inspiring work led by Bruno Latour (Latour and Woolgar 1979; Latour 1983; 1987; but see Rix *et al.* 1991) has shown that anything produced in a laboratory is the result of complex negotiations among different *actants*, and that instruments are part of these. Latour's analysis, based on a two-year 'ethnographic' study of the functioning of a laboratory, demonstrated that knowledge is not acquired but *constructed*, and that nonhuman elements such as artefacts play an important role in this process. Furthermore, laboratory studies showed that tools and equipment, and even scientific facts, are not only technically produced realities, but also entities that are symbolically and politically affected. The concept of *constructionism* thus became crucial, and the construction of science appeared as a result of countless negotiations inside and outside the laboratory (Knorr Cetina 1981; Bijker *et al.* 1987; Sturchio 1988; Knorr Cetina 1994). This set of ideas has recently been

proposed as the epistemological basis to integrate scientific analyses and archaeological theory (Jones 2002; 2004).

The analytical model that constructionism advocates for the study of modern laboratories is, in fact, very similar to that offered by the *chaîne opératoire* approach to material culture. Significantly, early material culture studies overemphasised the role of technical and material constraints in their interpretation of past activities, and had then to balance the picture by focussing on human agency (Dobres 2000; Dobres and Robb 2000; Gardner 2004), whilst traditional science and technology studies were exaggerating the role of humans and human needs, and had then to stress the active role of instruments and artefacts (Rabkin 1987; Rabkin 1993; Bijker 1994). Both approaches were somewhat myopic in their beginnings: the former had materials as sources of information; the latter, people and the books written by them – hence their respective biases. Fortunately, we have now both material and written sources to study early chymistry. If adequately exploited, combined and contrasted, they may render a more balanced view.

3.7. Summary

In this chapter, some limitations of traditional approaches to the history of alchemy and chemistry, particularly their excessive emphasis on individuals and their ideas, have been outlined. Subsequently, the vital role of instruments, experiments and laboratories has been argued. It was noted that some historical studies on early laboratories and archaeological studies of chymistry remains have started to redress this imbalance. Nonetheless, the lack of a solid theoretical framework and research strategies has led to a corpus of fragmentary works, rather than a coherent historical reconstruction.

Two theoretical models to solve this have been presented, one borrowed from material culture studies, another one from science and technology studies. None of them had been applied to chymistry, and both of them appear valid insofar as they complement each other. Whether we call it 'deconstructing' laboratory activities or 'constructing' a *chaîne opératoire*, the crucial aspect is striking a balance between constraints and choices, between the role of things and the role of humans, between technology and science. This thesis departs from scientific archaeology, but will bear in mind this wider framework to which it aims to contribute.

Not long ago, most archaeometric studies were still accused of "the lack of a common theoretical umbrella" (De Atley and Bishop 1991: 376) that could enable communication amongst specialists from different backgrounds addressing similar problems with dissimilar methodologies. From the beginning, I have explicit and deliberately opened my umbrella, hoping that it is big and flexible enough to cover many of us. It is my purpose to keep it open throughout.

4. Materials

"What profit is there of curious speculations, which doe not lead to real experiments? To what ende serves Theorie, if not applicable unto practice? And as this holds in all cases, so more especially it is true in Pyrotechny, *of which the whole would be vain and useless, unless the Theoremes of it were demonstrable practically by the Fire."*

George Starkey (1628-1655)

The laboratory equipment recovered in Oberstockstall constitutes the chief foundation and study object of this thesis. As this project evolved, however, wider questions emerged which required further investigation of archaeological remains from

Figure 18. Map of Europe in the 16th century (and outline of America, inset) showing the archaeological sites from which samples for analysis were obtained. The numbers refer to the sites in the same order as they will be discussed in the text: 1. Oberstockstall, Austria; 2. Obernzell, Germany; 3. Oxford, UK; 4. Großalmerode, Germany; 5. London, UK; 6. Jamestown, USA; 7. Porto, Portugal; 8. Burgsteinfurt, Germany; 9. Zwickau, Germany; 10. Cologne, Germany (after Shepherd 1923, modified).

different sites across the world (fig. 18). This section presents an introduction to all the sites from which samples were obtained for scientific analysis, in the same order as they will be discussed in the text.

4.1. Oberstockstall (Lower Austria)

Oberstockstall is an estate in the village of Kirchberg am Wagram, a renowned wine-growing region of Lower Austria, about 40 km to the West of Vienna. The site in its current condition is dominated by a large manor house or *Schloß*, constructed in the 16th century, attached to a 14th-century building with an adjoining chapel (fig. 19). This chapel underwent refurbishment in the mid-16th-century, which led to the annexation of a small room or sacristy in the back of the nave (fig. 20). A small slot in the wall of this room still allows a view of the altar (fig. 4, p. 34). In 1980, the owner of the building noticed a shallow depression on the floor of this sacristy. Concerned about the stability of the building, and further motivated by rumours about treasure buried in his property, he started digging in order to find out what lay beneath. Soon he realised that the treasure was different from what he expected, and gave notice to local authorities. This was the beginning of a historical and archaeological project still ongoing today.

Figure 19. The Oberstockstall chapel and adjoining buildings in early 2003.

In the course of two archaeological campaigns carried out in 1980 and 1993/4 (von Osten 1998), Sigrid von Osten identified what seemed to be a waste pit where about a thousand artefacts of ceramic and glass had been accumulated over a relatively short period (figs. 21-22). The abundance and variety of finds, as well as their state of preservation, were remarkable. The peculiar shapes of many of them clearly indicated that the assemblage constituted a collection of chemical equipment of some sort, together with residues of high-temperature operations such as slag, molten ceramics, charcoal and ashes, in addition to some other remains of metal, textile, leather and bone.

As the excavation developed, the vestiges of two furnaces were also identified[9]. What followed was the gigantic job of finding matching sherds amongst several thousands, and slowly re-assembling and cataloguing the different specialised instruments (see below, pp. 100-101). In the meantime, the deposition of the assemblage could be dated quite precisely with the aid of a coin, some inscriptions and dendrochronological research, to the period between 1548 and 1580/90, although the variability in the wares present suggests the possibility that some utensils could be older (von Osten 1998: 14-95). Specialist reports were obtained on the bone (Kanelutti 1998), wood (Cichocki 1998) and mineralogical remains (Thalhammer 1998), as well as preliminary scientific studies of the cupels (Rehren 1998) and other metallurgical residues (Sperl 1998). In the meantime, the comparison of the archaeological artefacts to those depicted in medieval and early modern sources allowed the identification of many pieces of equipment, and a preliminary assessment of their uses (Soukup and von Osten 1991; 1992; Soukup and Mayer 1997; von Osten 1998). A more ambitious project of scientific analyses emerged (Soukup and von Osten 1991; 1992; Soukup *et al.* 1993), but not more than a handful of analytical results of this project have been published (Soukup and Mayer 1997).

Parallel to the archaeological and archaeometric work, von Osten undertook a thorough review of the written documents relating to the site. It was possible to suggest that the likely masters of the laboratory were, successively but overlapping, two pairs of brothers: Christoph von Trenbach, Urban von Trenbach, Viktor August Fugger and Sigmund Friedrich Fugger, all of them priests with important ecclesiastic positions – two of them were bishops – who lived at this manor house (von Osten and Soukup 1992; von Osten 1998: 91-95). Von Osten's and further historical research by Soukup and Mayer (1997: 5-40) have added detailed information about their biographies and interests, including relevant information regarding their family relationships with important humanists and entrepreneurs of the time, ranging from apothecaries through iatrochemists to mining barons, and indirectly reaching up to the uncatalogable Paracelsus.

Nowadays, the *Museum Der Alchemist (Altes Rathaus)* of Kirchberg am Wagram serves as a permanent exhibition and study centre for the collection. Specific details of the samples selected for scientific analysis are given in the next chapter.

[9] Furthermore, a well-preserved Bronze Age burial was discovered, valuable but obviously irrelevant for the present study.

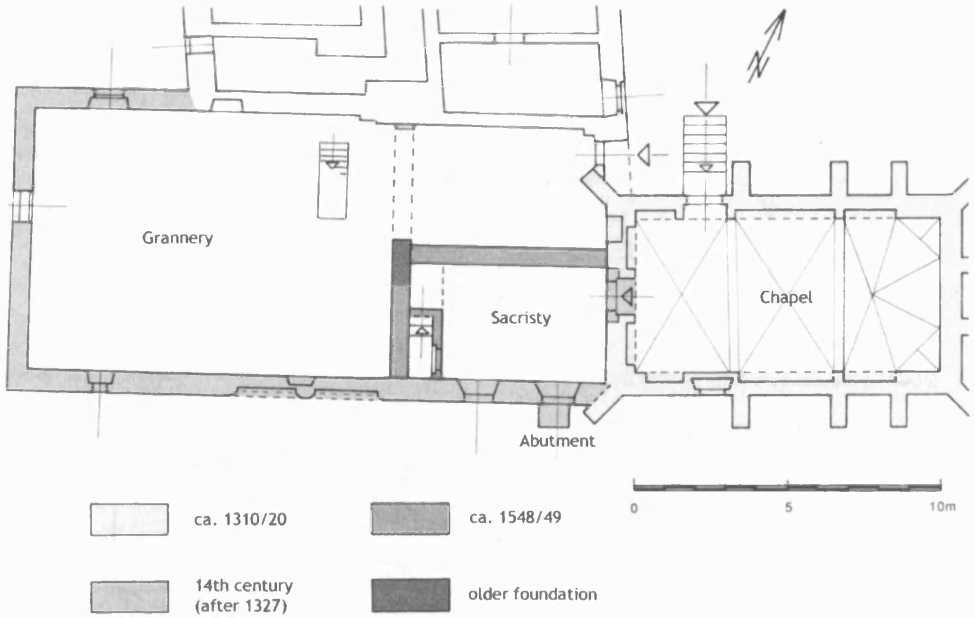


Figure 20. Plan of the chapel in Schloß Oberstockstall, and adjoining buildings (after von Osten 1998: 17, fig. 5, modified).

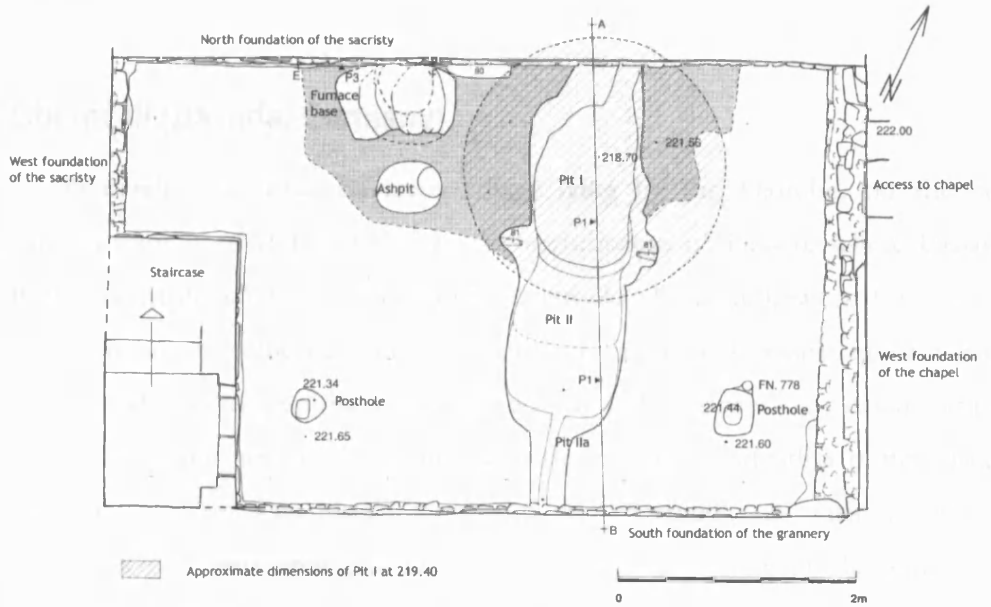


Figure 21. Plan of the excavations at the sacristy, with indication of the main features (after von Osten 1998: 22, fig. 7, modified).

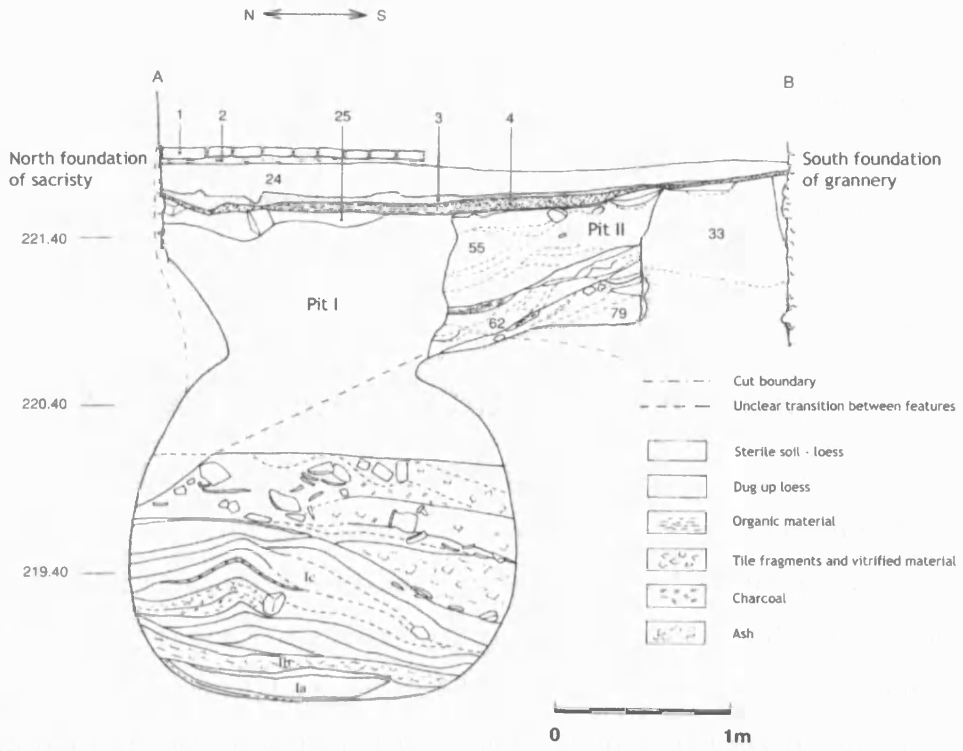


Figure 22. Profile of the pit where most of the laboratory equipment was discovered. Points A and B are marked in the plan in fig. 21 (after von Osten 1998: 20, fig. 5, modified).

4.2. Obernzell (Bavaria, Germany)

Obernzell is a small Bavarian village lying by the Danube, on the current German-Austrian border. It belongs to the archdiocese of Passau, where Urban von Trenbach, one of the chymists working in Oberstockstall, ruled between 1561 and 1598. Passau in general, and Obernzell in particular, were renowned in early modern times as large-scale producers of graphitic crucibles and other black wares[10]. As discussed later in this thesis, there were reasons to suspect that the crucibles and other pottery found in Oberstockstall might have been produced in Obernzell. A visit to the local *Keramikmuseum* was thus arranged, in order to investigate these productions in more detail and collect samples for comparative purposes.

Rudolf Hammel, ex-mayor of Obernzell and long-time researcher of the local pottery productions, kindly led the author's visit to the museum and supplied some samples from his private collection. Unfortunately, no precise archaeological contexts or

[10] Historical and archaeological details of the production of pottery in Obernzell are given in chapter 7, and further discussed in subsequent chapters.

dates for these are available, since they were collected during building works at a time when the recovery and safeguard of archaeological remains was unsystematic. However, based on Hammel's familiarity with the history of local productions, these can be estimated to date to the 16th or 17th century. The samples include one graphitic crucible and several fragments of black pottery, two of them stamped, as well as a lump of clay collected locally.

4.3. Old Ashmolean Laboratory (Oxford, United Kingdom)

Oxford's Ashmolean Museum was erected between 1679 and 1683. The underlying inspiration was Francis Bacon's utopian idea of 'Solomon's House', named after the Old Testament King Solomon, renowned for his intelligence and judgement, and mythical promoter of a temple inspired by Wisdom and designed to represent a model of perfection (cf. Lima and da Silva 2003: 29-37). To many of Bacon's readers, it seemed that the actual creation of a Solomon's House could give rise to a new era in the relationship between man and nature and, by extension, in the relationships among men, and between man and God (Bennett *et al.* 2000: 11-23).

Supported by the local University, which was ambitioning the incorporation of experimental natural philosophy in its curricula, this project materialised as a three-storey building in the heart of the city, which provided an exhibition room for Elias Ashmole's collection of rarities, a lecture room, a library and a laboratory. The combination of teaching, practical demonstrations and applied research was so rare that it astonished most contemporaries. The basement laboratory was described as "perchance one of the most beautiful and useful in the world, furnished with all sorts of furnaces and all other necessary materials in order to use and practice" (Chamberlayne 1684: 327; as cited in Bennett *et al.* 2000: 21). Here, the famous Robert Plot lectured and demonstrated as the first Professor of Chemistry at the University of Oxford, and the first Keeper of the Ashmolean Museum. He was aided by Christopher Whilte, who had learned his practical skills from Peter Stahl and Robert Boyle (Bennett *et al.* 2000: 11-23).

In 1781, when Martin Wall was appointed to a new Readership in Chemistry, the laboratory was renovated, and unwanted material including bones used for teaching anatomy, chemical vessels and domestic refuse were disposed of and covered by new limestone paving slabs. These are probably the archaeological remains discovered over two hundred years later, during refurbishment works of the Museum (site code

MHSO98/68). The ensuing archaeological excavation suggested that the entire basement terrace had been used as a dump, where clusters of human and animal bone, pottery, clay tobacco pipes, glass and ceramic chemical vessels had been discarded. Following this interpretation, it is important to emphasise that the materials recovered were those once regarded as no longer useful, and the original laboratory must have been much richer than shown by these remains. The dates for the entire assemblage were based on the domestic pottery, which showed a chronological span covering much of the 18th century, with a likely deposition date of *ca.* 1780 (Hull 2003; Bennett *et al.* 2000).

The chemical equipment included at least 18 round, flat-bottomed, open-mouthed crucibles of different sizes, 5 triangular crucibles, 2 oblong crucibles with relatively closed mouths, 2 retorts, 2 flasks, and a fragment of a lid. Details of the original characterisation of the ceramic fabrics will be presented later in this thesis, together with new information that helps correct misguided interpretations of the original report. As to the traces of use, preliminary XRF analyses by Chris Salter (Hull 2003: 11-13; Bennett *et al.* 2000: 31-47) showed relatively low metal and metal-oxide signals in the crucibles, in spite of their obvious distortion by high-temperature use. Still, based on these analyses, it was possible to suggest some of the likely operations carried out with this equipment, which included: the sublimation of lead and/or zinc, the preparation of lead silicate glazes, and work on sulphur or sulphates. The presence of barium sulphate and strontium sulphate was tentatively related to the production of coloured effects for fireworks. In addition, traces of mercury and antimony were detected in some vessels.

The Old Ashmolean is now house to the Museum of the History of Science, where part of this collection is exhibited. The archaeological remains and related archive institutionally belong to the Ashmolean Museum, which has moved to a new location.

4.4. Großalmerode (Hesse, Germany)

Großalmerode is the best-known crucible production centre in the region of Hesse. Since the Middle Ages, Hessian crucibles were manufactured in large quantities and exported across the world. The quality of the Hessian wares appears highly regarded

in several historical sources, to the extent of referring to their outstanding performance as a 'mystery'[11].

As the analytical work proceeded, it was realised that several crucibles from different sites had been produced with the same raw materials and following a very similar recipe, which raised the hypothesis of a Hessian provenance. Reference samples of crucibles from Hesse became thus necessary in order to ascertain this suspicion, and also to allow a more comprehensive picture of the production of crucibles in the Renaissance. Two crucible sherds were kindly made available by Ian Freestone, then of the British Museum, who had received them from Hans-Georg Stephan, of the University of Göttingen. They are undated surface finds.

4.5. Cripplegate Buildings (London, United Kingdom)

In the aftermath of World War II, William Francis Grimes, then director of the Museum of London, excavated parts of the Roman fort at Cripplegate. In the 1990s, an assessment of the material by John Shepherd revealed the presence of a well-dated assemblage from the early 17th century infilling of the city ditch, which included indicators of a variety of metalworking processes (Bayley 2003).

The metallurgical remains comprise approximately thirty crucibles of different sizes (from 50 up to over 200 mm high), most of them round with flat bases and open mouths, but including a few flat-bottomed triangular vessels and some globular ones. In addition, some ceramic scorifiers and one bone ash cupel are present. Finally, some wheel-thrown, redware bottles were identified as cucurbits, the long neck being made separately and luted onto the body of the vessel. XRF analyses of a number of samples were carried out by Justine Bayley (2003), who suggested that the biggest crucibles had been used for melting heavily leaded bronzes containing arsenic and/or antimony, whilst the smaller ones were related to the melting and assaying or refining of silver. The scorifiers and cupel also pointed in this direction. Finally, a red, powdery deposit of iron oxides left in several cucurbits was interpreted as a by-product of the distillation of nitric acid from a mixture of vitriol and saltpetre, thus indicating that the parting of gold from silver may have been conducted there too.

[11] The history and archaeology of crucible production in Hesse, as well as the 'mysterious' quality of the vessels, are addressed in chapter 8 and further discussed in the rest of the thesis thereon.

According to historical records, diverse metalworkers operated in the Cripplegate area during the Middle Ages, and the main cluster of goldsmiths in the City was precisely located in the vicinity of the excavated area. The archaeological remains appear thus related to goldsmithing activities (Bayley 2003: 8-9).

The assemblage from the Cripplegate Buildings is deposited with the London Archaeological Archive and Research Centre (LAARC), with the identification code WFG 18. For the present thesis, six sherds have been selected, representing optically different fabrics and formal types, and generally showing little traces of use.

4.6. Jamestown (Virginia, United States of America)

Located on the Chesapeake Bay, by the James River, Jamestown is often dubbed the 'birthplace of America', as it constitutes the first permanent English settlement in the New World. Its history starts in 1606, when the English king James I granted a charter to a group of entrepreneurs, the Virginia Company, to establish a settlement in that region of North America. Over one hundred men left London and sailed to Virginia with the major aims of settling the land, finding gold and a water route to the East.

In the years following their arrival in 1607, famine, disease and continuing attacks by the natives caused numerous casualties amongst the Englishmen, who had to build a fort and seek strategies for a peaceful co-existence and a productive settlement. The leadership of Captain John Smith saved the colony from dissolving completely, although difficult times were still ahead. Progressively, the relationship between natives and colonisers improved, as marked by the historic wedding of the Indian Pocahontas to the English John Rolfe in 1614. The systematic mismanagement by the Virginia Company, however, led the king to revoke the charter, and Virginia became a crown colony in 1624. Jamestown remained the capital of Virginia until the major fire of 1698. The capital was then moved to Williamsburg, and Jamestown was abandoned.

Since 1994, the Jamestown Rediscovery project, under patronage of the Association for the Preservation of Virginia Antiquities (APVA) and scientific direction by William Kelso, has excavated an important part of the Jamestown settlement, thus unearthing important episodes of the history of the colony (Luccketti *et al.* 1994; Straube and Luccketti 1996). The excavation has recovered considerable amounts of industrial waste, including dozens of beaker-shaped and triangular crucibles of different sizes, as

well as equipment for wet chemical operations, many of which seem to have never been used. Historical records attest the presence of apothecaries, refiners, glassmakers, blacksmiths and jewellers in Jamestown. Some of the crucibles, with clear residues of glass making activities, have been related to a "tryal of glasse" carried out by German glassmakers in 1607.

As far as metallurgy is concerned, it is known that the natives valued red copper, which they obtained as pendants and trinkets from the colonisers, in exchange for food. Recent research indicates that these copper artefacts were probably offcuts of copper sheet brought from England without much further processing (C. Hudgins, pers. comm. 2003). Nevertheless, the presence of metallic and submetallic deposits within some of the crucibles clearly suggest that high-temperature metallurgical operations also took place at the settlement. In this thesis, some of these crucible remains are analysed for the first time. They comprise a small triangular vessel containing a thick, black, glassy slag layer; a crucible fragment with some adhering metallic prills; a fragment of a big, beaker-shaped crucible; and a loose metal prill. They were made available by Carter Hudgins on behalf of the APVA.

4.7. Casa da Moeda (Porto Mint, Portugal)

The next archaeological site represented in this thesis is in the lower part of Porto city, by the Duero River in Northern Portugal, the neighbourhood housing industrial and commercial activities since the Late Middle Ages. The estate now occupied by the historic building known as *Casa do Infante* covers what used to be the *Alfândega Velha* ('Old Customs') and the *Casa da Moeda* ('Mint House'). Archaeological excavations at this site, coupled with historical research in the local archive, have allowed an insight into the organisation of the production process from the purchase of metal batches through assaying and refining activities and up to the minting of different coin types from metal sheets (Dordio *et al.* 1997; Lopes *et al.* 2000).

The house served as a mint from the 14th to the 16th century, with a later period of activity in the late 18th century. Differential concentrations of industrial remains within the site have allowed the identification of specific areas of activity such as the assayers' bench or the minting office, where the coins were struck. However, scientific analyses will be required in order to reconstruct these processes more precisely.

Over 100 bone ash cupels were recorded, together with 21 triangular and 13 conical crucibles (Dordio *et al.* 1997; Lopes *et al.* 2000). The assemblage was macroscopically examined by the author during a visit to Porto in 2003, and a wider analytical programme is under development. For the purpose of the present thesis, however, it was considered that one sample would suffice. A fragment of a triangular crucible was made available by Paulo Dordio, director of the excavations. Unfortunately, it lacks a clear stratigraphic context. As requested by the author, this sample was selected for its apparent unused condition.

4.8. Burgsteinfurt (North Rhine-Westphalia, Germany)

The Burgsteinfurt crucibles are from an unknown archaeological context. Most likely, they were excavated or found during building works somewhere in Burgsteinfurt/Münsterland in the second half of the 20th century and kept at the town's archaeological archive; at some point in time they were given to Professor Gerd Weisgerber of the Deutsches Bergbau-Museum in Bochum, who then passed them on, in the mid 1990s, to Thilo Rehren of the Museum's Institut für Archäometallurgie for analysis. Due to their lack of documented archaeological context they were deemed suitable for invasive sampling in order to retrieve as much technical information as possible, without harming a registered object. Unfortunately, no proper documentation of the various transactions has been kept, and no further details, such as tentative dates of use or deposition and related other finds, are known.

Four triangular crucibles were made available to the author of this thesis, of which only three were analysed invasively, given that the fourth one was complete and optically identical to one of the others.

4.9. Zwickau (Saxony, Germany)

The archaeological material from Zwickau was recovered during a rescue excavation directed by Jens Beutmann, and carried out within the medieval area of the city during 1998 (Beutmann *et al.* 2000). In a 7x7x1 m pit, some 3000 fragments of technical ceramic were found, together with some slag, clay moulds and furnace lining. Their deposition was closely dated stratigraphically and by dendrochronology to 1465-1491. The identification code for the assemblage is Z-29/234.

The crucible remains fall within three categories: big oblong pots, dome-shaped lids, and small triangular crucibles. They were analysed within another research programme focussed on the evolution of brass production, and interpreted as remains of a brass making workshop. The big crucibles would have been used with the lids for the cementation of brass from raw materials, whereas the small ones served for the melting and refining of the alloy prior to casting (Martinón-Torres 2001; Martinón-Torres and Rehren 2002).

No analyses of the Zwickau material have been carried out specifically for this thesis. However, the presence of triangular crucibles, which were previously analysed following the standard methodology described here (chapter 5), provided a sound basis for comparisons. That is the reason why the analytical results on the vessels from Zwickau will be discussed here again as part of a more comprehensive picture.

4.10. Weyerstraße, Cologne (North Rhine-Westphalia, Germany)

The material from Weyerstraße was acquired by the Cologne *Stadtmuseum* in 1966 and is known as *Alchemisten-Werkstatt* ('the alchemist's workshop'). Full publication of the archaeological information and the scientific analyses of part of the assemblage, carried out at the Deutsches Bergbau-Museum in Bochum (identification code D-94), is still pending, although a preliminary assessment after the archaeometric study has already been published (Rehren 1996a). The collection comprises a wide range of equipment used for both wet and dry chemical operations, including numerous distillation vessels of glass and ceramic, as well as beaker-shaped and triangular crucibles, scorifiers, shallow trays with a spout, one cupel, a rectangular lead vessel, and two antimonite cakes.

Earlier (unpublished) studies suggested that these could constitute the remains of a glass and/or glaze making workshop, but a more recent approach, focussed on the equipment and residues from high-temperature operations, refuted that interpretation and determined that these were more likely to represent a goldsmith's workshop. This conclusion was based on the clear evidence of refining of noble metals and gold/silver parting, together with the recycling of mercury gilding scrap (Rehren 1996a; Rehren 1996b).

The study of the residues within these vessels proved very informative of the range of reactions carried out in a Renaissance metallurgical workshop, while it posed

wider questions concerning the relationship between metallurgy and analytical chemistry in early modern Europe, some of which are addressed in this thesis. A comprehensive approach to the manufacture and technical standard of the instruments themselves will undeniably furnish more interesting information in this regard. For the present work, only one mounted specimen including ceramic material was obtained. It consists of a fragment of a rim attached to a lump of lute and containing several metal prills. Its fragmentary condition does not allow a definite identification of the main vessel type, which might be either a triangular crucible or a scorifier.

4.11. Summary

Although the laboratory in Oberstockstall constitutes the main case study addressed in this thesis, wider questions posed in the introduction, and further discussed later, made it pertinent to select a wider range of materials from comparative contexts. The most prominent factors behind these choices were the identity of the tools and techniques used for a wide range of high-temperature activities, and the likely existence of specialised production and trade of technical ceramics across the early modern world.

In terms of geographic distribution, special interest is placed in the area embraced by the Holy Roman Empire in the 16th century, given that this is the broader area contextualising Oberstockstall, and also because this is the region housing the main producers of crucibles and witnessing the most important developments in chymistry and metallurgy. However, samples from as far as Britain, Portugal or even North America are also studied, in order to obtain a more comprehensive picture of the distribution of these instruments and practices.

As concerns the immediate contexts of utilisation of the crucibles studied, the author has attempted to cover as large a range as possible, engaging the many facets of what has been defined above as 'chymistry'. Thus, not only crucible production sites are represented, but also remains from laboratories, goldsmiths' workshops, ore assaying, bronze and brass metallurgy, and a mint. Additional reference will be made to glass and glaze preparation by resorting to published studies.

5. Methodologies

"Habe ich viel gelesen/ viel gehört/ viel erfahren/ viel probirt/ viel laborirt/ viel speculirt/ bin auch mit viel gelehrten Leute umgangen."
[I have read much, listened much, experienced much, made many trials, laboured much, speculated much, and I have associated with many learned people.]
Johann Joachim Becher, Psychosophia (1635-1682)

5.1. Making choices: sampling strategy and analytical techniques

A foremost criterion when deciding the sampling strategy and the analytical techniques was ensuring that the results generated would contribute to the aims and objectives previously defined. The type of data, analytical sensitivity, precision and accuracy required were determined beforehand. In addition, among the various instruments available to each particular end, those requiring less invasive sampling and allowing replication were selected. Another factor observed was the methodological consistency with previous studies in the field, to facilitate the inter-comparison of results. In any case, some flexibility in the methodological programme was allowed, expecting that new queries would arise as the results started to come out.

Since the project aims at the full comparative characterisation of technical ceramics and a preliminary approach to chemical operations, all three types of mineralogical, chemical and physical information were considered of interest. The manufacture of the vessels, their material properties and performance during use could thus be more thoroughly investigated. Furthermore, a comprehensive data set shall facilitate comparisons among the samples studied here and those (to be) studied elsewhere. The particular justification and capabilities of each technique are detailed under the relevant subheadings.

For the Oberstockstall case study, the sample selection went through various stages. Firstly, after restoring, numbering, drawing and formally classifying the entire collection, Sigrid von Osten submitted a selection of about one hundred artefacts or fragments. Thilo Rehren (1998) carried out a preliminary investigation of the bone ash

cupels by x-ray diffraction (XRD), scanning electron microscopy (SEM) and inductively coupled plasma – optical emission spectrometry (ICP-OES), and kept the material for future enquiry. When this PhD project started in October 2001, all the available samples were optically inspected in detail, and many of them non-invasively analysed by energy dispersive X-ray fluorescence (ED-XRF), before undertaking a detailed examination of polished samples of a number of them by optical microscopy and scanning electron microscopy – energy dispersive spectrometry (SEM-EDS). These analyses ran parallel to the widescale literature review discussed in previous chapters.

Once the assemblage was broadly characterised and the research questions better defined, a study visit to the *Museum Der Alchemist* in Kirchberg am Wagram (Austria) was planned for early 2003. The whole assemblage was then re-examined in the light of the new information and queries, and most of the artefacts and other debris were photographed. It was important to obtain a sample that was suitably representative in order to be able to draw significant conclusions without wasting resources or generating too many redundant data (Orton 2000) – although foreseeing data replication in some cases. The continuing availability of the original sample provided by von Osten allowed an 'adaptive sampling strategy', where the questions to be answered were refined in the light of preliminary analysis. After this, it was resolved that the main focus of this stage of research would be the ceramic material, obviously related to the various uses in order to assess performance, but leaving the detailed characterisation of the chemical reactions for a future stage[12]. It was decided to select a 'stratified sample', *i.e.* one where all types and features are represented but not pointlessly repeated. Under more precise criteria, another forty artefact fragments were selected during the visit to the Museum, including at least one sherd of each of the ceramic types optically identified, as well as several fragments of the many crucibles and scorifiers, since preliminary analyses indicated a wider variability of pastes within these artefact types. Finally, even though they would not be used for the present thesis, twenty samples of residues were scrapped off ceramic artefacts, up to two cubic millimetres each, and duly preserved for future investigation.

A brief note should be made on the use of invasive analyses. Archaeological remains are common heritage, and one of the main reasons why they are recovered and preserved is to serve as sources of information about the past. It is an archaeologists'

[12] At the moment of submitting this thesis, Aude Mongiatti is beginning her PhD project, which will address this dimension of the Oberstockstall assemblage in more detail.

duty to exploit these sources as far as possible (cf. Tite 2002; Rehren 2002b). From this point of view, albeit the author is committed to inflict the least possible alteration to archaeological materials during study, it was considered that invasive sub-sampling was justified in some instances, as this was the only way of obtaining relevant data that would otherwise remain unknown. When this was the case, the subsamples were removed trying to embrace the largest amount of information with the minimum damage to the sample. For example, in a cross-section of a crucible sherd, the cut was performed parallel to a fractured edge and in such a way that, in addition to the fabric itself, vitrification or slag remains could also appear in the mounted specimen. When more than one sample of the same material existed, the smallest and/or the one containing more information was selected. No complete or unique artefacts were sub-sampled. All the sampling points were indicated in sketch drawings and photographs of the artefacts.

Most of the samples from Oberstockstall possess three reference codes: a find code (given in the field), an inventory number (given after restoration), and a fabric type code (given after optically discriminating the fabrics – see von Osten 1998). Since the inventory number is physically imprinted in the samples themselves, this was the one to be used at the laboratory in London. All the specimens removed were labelled as follows:

OB 560/s2 MED BLACK CRUC Jan 2001 MMT

In this label, OB stands for Oberstockstall, 560 is the inventory number, s2 denotes the specimen number (anticipating cases where more than one specimen were removed from the same sherd), MED BLACK CRUC is a brief means of identifying a medium-sized black crucible, Jan 2001 is the date and MMT are the initials of the person who mounted the specimen. The few analysed sherds that lacked an identification number were labelled OB n001 and so forth. The same procedure was followed when preparing specimens from other sites, always keeping the original site identification code whenever this was available.

The type of specimen used for both optical and electron microscopy was a standard cross-section mounted in epoxy resin, ground and polished down to one micron particle size.

5.2. A note on the analysis of ceramic fabrics

Fabric can be defined as "the composition of a fired ceramic, including clay, inclusions, and pores and excluding surface treatment; often used synonymously with *body*, *paste*, or *ware*" (Rice 1987: 476). The study of this 'internal' dimension, completed by 'external' features such as the morphology and surface finish of the artefacts, provides rich information for the comparative characterisation of ceramics. The physical, chemical and mineralogical characteristics of any ceramic fabric are determined by "(i) the natural composition of the raw material(s); (ii) the actions taken by the potter in creating the clay mix (...); (iii) the firing atmosphere and temperature [to which 'duration' could be added]; and (iv) the use and post-depositional environment of the vessels" (Orton *et al.* 1993: 132). Traces of all these stages are often kept in the fabric. Accordingly, fabric analysis aids the characterisation, sourcing and provenancing of vessels; the study of fabrication techniques, including manufacture and firing; and, in some cases, dating.

Leaving dating techniques aside, two main approaches to the study of ceramic fabrics are available to the archaeologist. The first one is based on the optical examination of ceramic sections using methods adapted from petrography; it is generally referred to as 'ceramic petrography' and focuses primarily on the study of 'textures', *i.e.* the identity, arrangement, size, shape and distribution of particles within the ceramic matrix (e.g. Freestone *et al.* 1982; Middleton and Freestone 1991; Freestone 1995; Middleton 1997). The other approach consists of the chemical analysis of pastes, and therefore is mostly concerned with the ceramic material itself, rather than the inclusions (e.g. Neff 1992). Both approaches are complementary and well established. Since this thesis aspires to constitute a comprehensive reference study, both have been combined here.

There is no clear agreement as regards the terminology used for describing ceramic fabrics, therefore it is necessary to demarcate the terms as used in this thesis (fig. 23). The term 'fabric' is used as defined above, to denote the ceramic material generally, including clay, inclusions, porosity and their interrelation. 'Matrix' is the relatively continuous phase made up by the

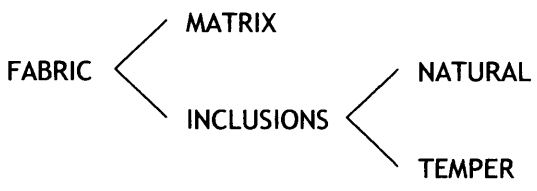


Figure 23. Diagram demarcating the basic terms used in the characterisation of ceramic fabrics.

vitrified clay and holding the fabric components together. 'Inclusions' are any discrete particles interspersed within the ceramic matrix but optically detached from it – they are thus part of the fabric, but not of the matrix. Lastly, the term 'temper' is reserved to name inclusions artificially added to the clay.

5.3. Optical microscopy

5.3.1. Stereoscopic microscopy of samples as received

Stereomicroscopes are valuable aids for three-dimensional observation and perception of depth and contrast. The stereoscopic microscopy facilitated the scrutiny of some relevant features such as external traces of manufacture on the vessels, the texture of different materials, or a primary assessment of the vitrification of ceramics as seen in fresh fractures. It was of use in deciding which specimens were to be mounted for detailed examination, and the sampling points. During the preparation of polished specimens, the stereomicroscope was routinely used to check the surface finish at different stages during grinding and polishing. The stereomicroscope used was a Wild Heerbrugg.

5.3.2. Reflected light microscopy of polished cross-sections

The applications of reflected light microscopy of archaeological ceramics are not very well documented, and in the literature it is sometimes assumed that this technique is of little use in microstructural analysis (Haynes 1984; Bousfield 1992). The optical microscopy of polished sections is not even considered in a recent handbook of archaeomineralogy (Rapp 2002). However, this technique provides helpful information on the topology and optical properties of the examined surface, and thus supplies the first clues for the understanding and classification of different materials.

Even though the use of transmitted light microscopy of thin sections would have facilitated the identification of mineral phases in ceramic fabrics – thereby allowing the definition of provenance groups and an assessment of the firing or use temperatures (cf. Maggetti 1982; Freestone 1995; Middleton 1997) –, it was decided that reflected light microscopy of thicker polished sections would be used. The basic reason behind this choice is that also slag and metal by-products adhering to most ceramics were to be studied, and the examination of these crystalline and glassy phases is more complete if

undertaken using the latter technique (Bachmann 1982; Scott 1991: 321). Besides, the microscopy of polished sections of crucibles had already been used by the author in a previous project (Martinón-Torres 2001) and proved useful. Furthermore, polished cross-sections could then be directly used in the SEM for the identification of mineral and glassy phases.

The examination of ceramic fabrics and slag by optical microscopy paid special attention to the following aspects: general appearance and reflectivity; interfaces and interaction between different phases; abundance, shape and distribution of porosity; nature, abundance (in percent by volume, vol%), size uniformity ('sorting'), distribution, and degree of sphericity and angularity of inclusions. Optical microscopy under polarised light (PL) was useful to study the interaction between metallurgical debris and ceramics, and to detect the presence of bright metallic phases such as silver-rich globules in lead-dominated phases, which are not always easy to identify in electron microscopy due to the different imaging principles. Intermetallic compounds, graphite or nonmetallic inclusions and other small particles with high reflectivity were also best observed under PL. With the polariser and the analyser fitted in the microscope – *i.e.* under cross-polarised reflected light (XPL) –, colour contrast allows a better examination of homogeneity and the orientation of ceramic matrix, as well as of the spatial orientation and distribution of the crystalline phases. Finally, the examination of some specimens under only 'slightly' polarised light (sXPL) was found useful in that translucent inclusions such as quartz grains are visible but the colour of the ceramic pastes is not removed (fig. 24).

The instrument used was a Leica DM LM. Photomicrographs were routinely taken of the specimens under PL and XPL, at 50x, 100x and 200x magnification, apart from other images of specific features.

Figure 24. Three different micrographs of the same area of a crucible fabric. From left to right, PL, XPL and sXPL (OX n001/s1, 50x, long axes represent ~2 mm).

5.4. Energy dispersive X-ray fluorescence spectrometry (ED-XRF)

X-ray fluorescence is the characteristic emission following the absorption of high-energy radiation by atoms in the X-ray region, *i.e.* in the inner shells of electrons or orbitals (K, L or M). This technique is therefore based on atomic inner shell processes. If an X-ray is directed to the sample material and hits an inner orbital electron at sufficient energy, this is ejected from the atom. Once an atom has lost a core level electron by the absorption of an X-ray photon, an electron from a higher shell refills the hole. In this process, energy is released which can either cause a third electron to leave the atom (Auger effect) or may be emitted as a high-energy, secondary, fluorescent X-ray (XRF). The energy of a fluorescent photon is determined by the quantum numbers of the initial and final electronic states and the atomic number of the atom. Accordingly, every element contained in the sample leaves its characteristic fingerprint or spectrum of XRF photons and, hence, can be identified through appropriate measurements. The XRF instrument triggers this process under controlled conditions, screens those signals and displays them on a spectrum where the position of each peak is characteristic to one particular element, and the area under that peak is relative to the amount of that element present in the sample (Dziunikowski 1989; Potts 1995; Pollard and Heron 1996; van Grieken and Markowicz 2002).

Two principal ways for the determination of the characteristic photons are possible: wavelength and energy dispersive systems. The former type mainly consists of an analyser crystal and a flow counter; the latter, of a semiconductor crystal. In any case, the results of both XRF systems can be interrelated by means of a simple equation that relates the energy E of a given X-ray photon (in keV) with its equivalent wavelength λ (in Å): $E=12.4/\lambda$ (Pollard and Heron 1996: 46).

Wavelength dispersive spectrometers (WD-XRF) are capable to analyse one characteristic line at a time with high resolution and good peak to background definition. The main disadvantage is the need of a sequential – *i.e.* element by element – analysis of the signal. Energy dispersive detectors (ED-XRF) use the semiconductor crystal to transform the incoming energy of a single photon into a well defined number of electron hole pairs, representing a characteristic amount of charge for every incoming photon. A spectroscopic amplifier and a multi channel analyser convert the charge signal to a numerical pointer and the photon is counted as an event of the appropriate energy in the computer. The complete spectrum may thus be analysed simultaneously.

In sum, ED-XRF is quicker and allows real-time identification of elements without the need of previous knowledge about the nature of the sample. Its main setback in comparison to WD-XRF, that is the usually higher detection limits, is being progressively overcome by the use of polarised X-rays (P)ED-XRF, which allow a significant background noise reduction in the spectrum (Veldhuijzen 2003). Another potential limitation of ED-XRF is the resolution of overlaps in the spectra between peaks corresponding to different elements. However, given that this instrument was only used for preliminary screening purposes and accurate quantification was not needed, peak overlaps did not constitute a major hindrance. Furthermore, given the precise analytical geometry required for WD-XRF analysis, its use would have demanded sample removal and preparation, *i.e.* exactly what we were trying to avoid.

The instrument at the Institute of Archaeology is a (P)ED-XRF Spectro XLab 2000. The computer software attached to it transforms the XRF signal in a spectrum, compares the peaks to those obtained from the analyses of certified reference materials ('standards'), and from this comparison it calculates the elemental concentrations in the sample. The analytical settings were those of the standard Spectro 'Oxides' method, which includes three polarising targets (Compton/Secondary Molybdenum, Barkla Scatter Aluminium Oxide and Barkla Scatter HOPG).

Three main factors led to the consideration of this instrument as ideal for the preliminary analysis of the surfaces of ceramics. Firstly, the possibility of avoiding invasive preparation by analysing samples as received, as most of them fitted in the instrument chamber. Secondly, the comparatively big diameter of the X-ray beam (~1.5 cm), which allows bulk analyses of relatively large areas. Thirdly, the relatively low penetration depth of the X-rays – only a few millimetres, depending principally on the average atomic weight of the elements in the sample and the energy of the exciting beam. By comparing the composition of inner and outer surfaces, one can estimate the inner contamination from use. Also an initial approach to the different fabrics' compositions could be obtained, even for those samples where the removal of specimens for further examination was not acceptable.

Since the samples analysed were neither flat nor homogenised – as required for accurate XRF analysis –, the results could only be semi-quantitative. In addition, no attempt to conjecture the proportions amongst elements originally contained in the vessels' charges would be made on the sole basis of XRF results, since the incorporation of the different metallic elements in the fabrics is conditioned by several factors that are

not easy to predict (Dungworth 2000). However, this non-invasive approach, coupled with the optical examination of the assemblage, helped define the first working compositional and usage typologies and directed further questions for this and forthcoming work.

5.5. Scanning electron microscopy – energy dispersive X-ray spectrometry (SEM-EDS)

The foundations of electron microscopy are explained in several handbooks (Potts 1995; Pollard and Heron 1996; Reed 1996; Watt 1997; Brandon and Kaplan 1999). Briefly, a scanning electron microscope (SEM) consists of four main components: a column with an electron gun that generates a beam of electrons, a specimen chamber where the electrons interact with the sample, some detectors that monitor the different signals that result from this interaction, and a viewing system that constructs an image from the detected signals. The basic principles behind the SEM are relatively simple: a tungsten filament is heated and emits an electron cloud, which is focussed into a beam and directed towards the specimen by means of an electromagnetic condenser. The interaction between the electrons and the sample atoms generates assorted signals, such as secondary electrons (SE, electrons from the sample surface itself, with energies up to 50 eV), back-scattered electrons (BSE, elastically scattered back from the incident beam, with energies above 50 eV), X-rays, light, heat, and even transmitted electrons that pass through the specimen. The incident beam scans over the sample line by line, and the relevant detector builds up a complete image.

Since the wavelength of the electrons is much smaller than the wavelength of light, the resolution and magnification capabilities of the SEM are much greater than those of the conventional compound light microscope, reaching down to ~5 nm. Furthermore, the SEM has a large depth of field, which allows a relatively large amount of the specimen to be in focus at one time. Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. For this work, the specimens used were the same polished cross-sections as examined by optical microscopy, after carbon-coating in a standard vacuum carbon sputter. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in materials characterisation.

The SEM instrument used for this research project is a Philips XL30 Environmental SEM (ESEM) with an INCA Oxford spectrometer package, housed at the Institute of Archaeology. It is equipped with both SE and BSE detectors. The factors affecting SE emission are the morphology of the surface, the beam energy and intensity and, to a lower extent, the density of the sample. On the contrary, the BSE intensity only depends on the average atomic number of the specimen. Combining both devices, one obtains a great amount of information on the topography and the compositional make-up of the specimens examined.

Lastly, the SEM used has attached both wavelength (WDS) and energy-dispersive (EDS) X-ray microanalysers, the principles of which have already been explained for the XRF. The EDS allows measurements in a scanning mode, where an area of up to ~2,5 cm^2 may be analysed, and also spot analyses of particles down to a few μm diameter, and was utilised routinely. The WDS was occasionally used for resolving peak overlaps in some spectra. In general, SEM-EDS is regarded as the easiest method – and sometimes the only one – for analysing microscopic samples, when the accurate detection of minor elements is not crucial. Further advantages of this technique are its non-destructiveness and the relatively limited requirements for specimen preparation.

The use of SEM-EDS was considered especially pertinent for the imaging capabilities mentioned above, which would help refine the characterisation of different ceramic materials and traces of use. Besides, the microanalysis package allowed a detailed chemical study of fabrics and slag remains. The elemental analysis of fabrics addressed matrices and inclusions separately, following the example established by pioneer works in the field of archaeological refractories (Tite *et al.* 1985; Freestone and Tite 1986; Freestone 1989; Bayley *et al.* 1991) and recently applied to more case studies (Martinón-Torres 2001; Martinón-Torres and Rehren 2002; Hauptmann *et al.* 2003). The composition of matrices was obtained as averages normalised to 100 percent by weight (wt%) of 5 to 10 measurements of homogeneous areas of ~70-100 by 100-150 μm made on one to two specimens of each sherd. This conventional area size was decided as it was the largest possible uniform matrix area not embracing big inclusions that could be analysed in most of the ceramic specimens. The composition of specific inclusions was then probed independently. A similar approach was adopted for the analysis of slag and metal remains – separating the analysis of individual phases such as metal globules or dendrites – although also bulk analyses of larger areas were undertaken.

Operating conditions for SEM-EDS data collection were as follows: working distance of 10 mm; accelerating voltage of 20 kV; spot size of 4.7 to 5.3 (INCA conventional units) and process time 5 or 6, corresponding to a detector deadtime of 25-40%; and livetime of 50 seconds. In order to permit consistent comparisons, images of all the specimens were routinely taken at 50x, 100x, 500x and 800x magnification, together with others of individual features at discretion.

Although the SEM instrument is calibrated with a comprehensive set of standards, accuracy and precision were routinely tested via the analysis of pellets of siliceous certified reference materials. These were made by mixing each of the powdered standards with epoxy resin and forming a pellet which was then cut, mounted and polished in the same way as the other ceramics. These 'standard specimens' were then analysed following the established methodology. In accuracy tests, analytical results compared to certified values showed coefficients of variation typically below 10% for all major element oxides except magnesia, with the error increasing for elements in minor concentrations, although usually remaining below 20%. Coefficients of variation in precision tests were typically below 5% for major, and up to 15% for minor, element oxides. Lower confidence limits were established at 0.5 wt%, but values below this limit are also given as indicative where considered relevant. When the routine tests showed higher variation ranges, the analyses were interrupted to run a detector conditioning programme, which habitually resulted in then obtaining satisfactory results.

All the chemical compositions presented in this thesis will be given as percents by weight (wt%), and the results normalised to 100%. Un-normalised totals inevitably were below 100% due to the inherent porosity of most samples and drift in beam intensity. However, the data quality control procedure described above allows us to trust the obtained results regardless.

5.6. Summary

The sample selection followed an adaptive strategy. After preliminary analyses leading to more precise questions, a stratified sample of the assemblage was selected, where all the main types of fabrics were represented. High-temperature ceramics were more heavily represented so as to allow a more detailed study of their material properties and performance characteristics.

ED-XRF was used for non-invasive analyses, mostly aimed at a preliminary assessment of compositional groups and traces of use. Subsequently, analyses used mostly optical microscopy and SEM-EDS of polished cross-sections. This was deemed the most suitable instrumental technique for this project, after a careful evaluation of factors such as the precision and accuracy required, the principle of minimum alteration of the archaeological materials, and consistency with previous work. Standard analytical protocols were established in order to allow systematic comparisons.

6. The laboratory equipment from Oberstockstall: technological choices and performance characteristics

"What most experimenters take for granted before they begin their experiments is infinitely more interesting than any results to which their experiments lead."
Norbert Wiener (1894-1964)

"Thou hast done nothing but pile up ambiguous words. Return, therefore, to the subject."
Turba Philosophorum (ca. 1200)

6.1. Introduction

The laboratory equipment recovered in Oberstockstall shows a great variety of formal types and visually different ceramic fabrics. A thorough formal organisation and a tentative macroscopic fabric classification have been offered by von Osten (1992; 1998) after a detailed optical examination. In general terms, at least four main groups of ceramic fabrics can be distinguished: unglazed buff/orange, green-glazed, orange/brown-glazed, and black wares. Additional fabric types are represented by building ceramics such as furnace bricks, and by the lute used for coating or sealing some vessels. Finally, crucibles, scorifiers and cupels form distinct categories, however the alteration of their appearance through their high-temperature use made it impossible to visually determine whether they

Figure 25. Selection of black wares from Oberstockstall. Scalebar is approx. 15 cm.

Figure 26. Some of the Obernzell stamps on the black wares found in Oberstockstall. Long axis of the stamp is ~3 cm (drawings by Sigrid von Osten).

Figure 27. Unglazed ceramic artefacts from Oberstockstall. Scalebar is approx. 15 cm.

Figure 28. Green-glazed equipment from Oberstockstall. Scalebar is approx. 15 cm.

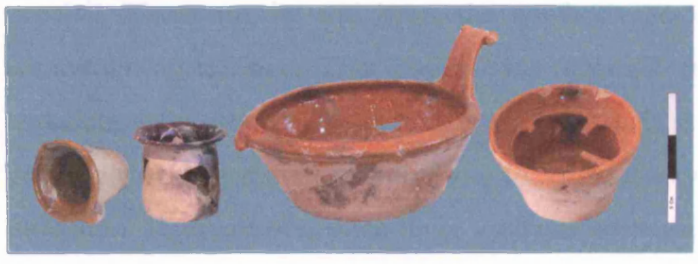


Figure 29. Brown- and orange-glazed ceramics from Oberstockstall. Scalebar is approx. 15 cm.

belong to one or more fabric types. To complete the picture, it should be mentioned that different types of glassware are present, including flasks, phials and distillation columns, although they will not be considered in this work.

Black wares normally comprise open pots of different sizes, up to 40 cm in height, oblong or cylindrical, with or without handles, with wide convex or flat rims that reach 40 cm in diameter (fig. 25). Many of these rims exhibit one or more stamps. They are shield-shaped and show variations of a similar exhibit one or more stamps. They are shield-shaped and show variations of a similar motif that combines equi-axed crosses with other segments and dots (fig. 26). Some also show simpler engraved marks, such as crosses, parallel segments or dots, on the rims and body surfaces. The more complex stamps have been identified as typical from Obernzell (Bavaria, present-day Germany), a well-known village producing and exporting black and other wares since late medieval times (von Osten 1998: 56; see also chapter 7). Unglazed ceramics of a buff to orange colour are represented by muffles, funnels, sieves, cucurbits, retorts and receivers used in distillation and sublimation operations, as well as a series of ceramic plugs probably used to adjust the air draft into the furnace (fig. 27). Internally green-glazed ceramics are used for a variety of chymical equipment, including distillation and sublimation capitals,

adopters and sieves (fig. 28). Finally, orange glazes appear mostly on bowls, pipkins and cylindrical pots, as well as more intricate types, possibly used in sandbath distillation (fig. 29).

The most characteristic artefact shapes have already been identified by von Osten (1992; 1998) whose work was later completed by Soukup and Mayer (1997). Based on some contemporary written sources, it was possible to offer a preliminary interpretation of the main applications of most of the pieces of equipment. In summary, apart from ordinary, less diagnostic shapes, von Osten (1998: 86) identified the following types: 4 alembics – one made of ceramic, for sublimation, 3 of glass, for distillation –; 2 tailed flasks (a third one is uncertain); 4 sieves of different shapes and materials; 13 'steamer domes', 5 funnels (which could as well have served as steamer domes), one distillation column, 6 'sandbath' instruments, 11 bulbous recipients and melting pots of different kinds, altogether 25 ceramic distillation receptacles, inclusive of retorts (complete and fragmentary), and besides 7 glass distillation receptacles and 14 glass phials; about 280 crucibles, 50 scorifiers, 100 cupels, and 13 reasonably complete muffles complete the inventory.

The analytical work presented here largely focuses on technical ceramics used for high-temperature reactions – namely crucibles, scorifiers and, to a lesser extent, cupels[13]. These are, by and large, the artefact types most heavily represented in the laboratory assemblage. This choice was directed not only by the higher relative abundance, but also by the fact that artefacts such as crucibles and scorifiers were so versatile that their mere ascription to a formal category tells very little about their actual utilisation. Furthermore, since these artefacts were used at very high temperatures and often containing corrosive metal oxides, their surfaces appear so distorted that it is difficult to attempt any grouping without scientific analyses. Moreover, their high-temperature use purports that more traces of the materials processed were left within the vessels and are thus susceptible of analytical identification. Finally, the aim to contribute to the long-standing discussion regarding the manufacture and supply of crucibles in post-medieval Europe and beyond was a further factor leading to this decision.

[13] The analytical examination of cupels specifically carried out for this thesis is constrained to one unused cupel and a lump of bone ash. However, data from previous work by Thilo Rehren (1998; Martinón-Torres and Rehren 2004b) will be discussed together with the new data in order to present a more comprehensive picture.

Of the other ceramics, only one or two samples of each broad fabric type (black, orange unglazed, green-glazed and orange-glazed wares) have been analysed. This was considered enough for a general characterisation of these visually rather uniform fabrics, which would be useful for comparative purposes within this assemblage, and may also be of use for future comparisons with similar formal types found elsewhere. Given the lower temperature and often aqueous nature of the reactions undertaken in these instruments, less traces of use are normally left in their surfaces. However, some still exhibit sediments of the substances processed. Analyses of a small number of these will be presented, although this does not constitute a focal point of this work.

6.2. The triangular crucibles

For this thesis, the term 'crucible' is used to designate a free standing, movable vessel, generally used for 'dry', *i.e.* high-temperature, operations (cf. Rehren 2003: 207) The triangular or 'laboratory' crucible is an icon of the Renaissance laboratory, and as such it appears depicted in innumerable paintings (see e.g. fig. 6, p. 37). This vessel constituted the basic tool for any small scale pyrotechnological reaction. As discussed later in this thesis, triangular crucibles were used in copper and brass metallurgy, for gold and silver working and in all sorts of assays. Assayers needed them, but also chymists of all kinds, apothecaries, physicians, coin minters, glass workers or even potters for glaze preparation (Cotter 1992: 265). This wide range of possible uses stresses the need for well-preserved archaeological contexts and analytical studies in order to identify both specific crucible types and activities.

High quality crucibles have been in demand for centuries, which explains the detailed recipes for making crucibles given in several treatises since the Middle Ages (e.g. Sisco and Smith 1949; Wyckoff 1967; Hawthorne and Smith 1979), and the fact that, already in the late 17th century, the reason behind the outstanding quality of the famous Hessian crucibles is still referred to as a "mystery" (Plot 1677: 250). Wider discussion concerning the production and consumption of crucibles in the post-medieval world will be presented towards the end of the thesis (chapters 7-10), drawing together the analytical results from several assemblages and substantial information from written sources. For this chapter, the focus is placed on the crucibles from Oberstockstall only.

As noted in chapter 3, the characterisation of the artefacts will use some analytical categories borrowed from material culture studies. The different features of the crucibles may be discussed as the result of different technological choices made at early stages of the *chaîne opératoire*, which conditioned the formal and material properties of the vessels, and thereby affected their performance characteristics (cf. Schiffer and Skibo 1997; Skibo and Schiffer 2001; Sillar 2003; Schiffer 2003).

Firstly, the formal properties of the vessels shall be presented, placing the emphasis on their condition prior to use. Then, their formal and material properties shall be discussed in the light of analytical measurements and relevant technical literature, considering the choices and decisions made by producers and users of the crucibles. The contexts where these crucibles were produced, traded and used shall

provide the clues to explore the reasons behind those choices: only by considering the performance of these vessels, both in the market and in the laboratory, can we assess the constraints that led the potters to manufacture them in a particular way, and the extent to which the different material properties observed were relevant to 16th-century potters and chymists. In this sense, reference will have to be made to the specific reactions carried out within some of the vessels. The last part of this section will present analytical details of some of the used crucibles, with a view to investigate their specific conditions of use, technical requirements and corresponding performance.

Figure 30. Group of triangular crucibles from Oberstockstall. Big scalebar is 15 cm, small one is 5 cm.

6.2.1. Characterisation

The design of all the crucibles recovered in Oberstockstall follows the same formal pattern: the base is circular and flat, and the body opens progressively to end in a triangular mouth (fig. 30). This characteristic shape was achieved by pushing inwards the rim of a (truncated) conical vessel. In most cases it is clear that the crucibles were wheel-thrown before the rim was folded inwards, as is especially noticeable in the bigger ones, which show parallel horizontal grooves on their

Figure 31. Some of the smallest crucibles.

surfaces (fig. 32). The formal standard of the almost three hundred crucibles is remarkable, and those within the same size ranges (see below) are often nearly identical. The height of the vessels tends to be roughly equal to the length of each side of the triangular mouth, although there are exceptions of both slimmer and wider ones. For a medium-sized 100 mm tall crucible, the body thickness is about 6 mm, tapering towards the rim and being thicker at the base. Smaller crucibles tend to have relatively smaller bases, which results in more open profiles. This is shown by the ratio between the side of the mouth and the base diameter, which is normally in the range 1.8-2.2 for crucibles up to 90 mm high, and between 1.4-1.8 for larger vessels.

Figure 32. Two views of a big crucible, where traits from the manufacture on the wheel can be noticed.

Sizes span from remarkably small vessels of just 16 mm in height (fig. 31) to large crucibles of up to 185 mm (fig. 32). Approximately 60% of them are stamped on their bottom with a symbol resembling a letter 'T'. Normally, the smallest crucibles do not show any stamp, the next size range has one, then there is a group with either one or two stamps, and the biggest ones always bear two (figs. 33-34), although there are some medium-sized crucibles without any stamp. Hence, whilst the number of

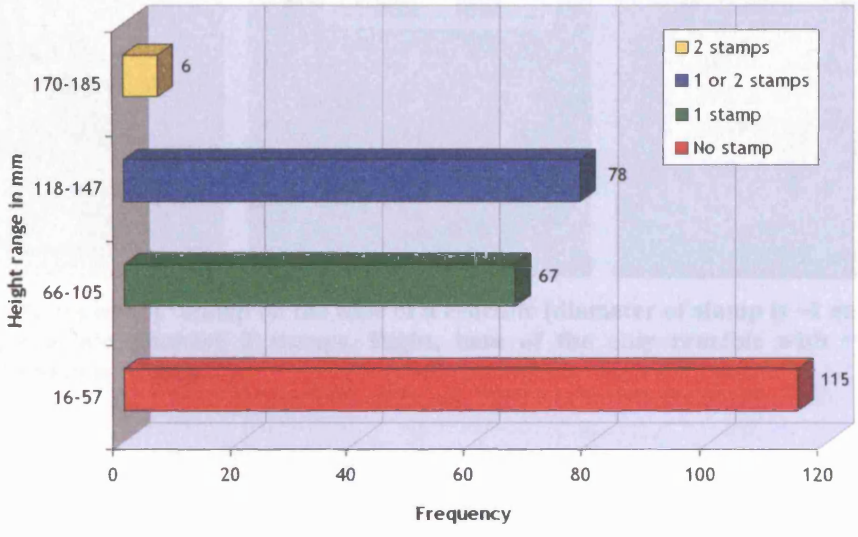


Figure 33. Bar chart representing the frequency distribution of different crucible sizes and the presence of 'T' stamps (after data from von Osten 1998: 41-42).

stamps could relate to the capacity or value of the crucibles, this criterion does not seem to have been followed very strictly. Variations of this stamp have been recorded in crucibles and other pottery types across the region spanning from Bavaria through Austria and up to Hungary, although the meaning of the symbol, or the production centre(s) that applied it, remain unclear (cf. Holl 1976 and section 7.5).

The group of smallest crucibles does not appear to meet the above formal standard so tightly (fig. 31). These were hand moulded, with the help of a round stick with a ~4 mm-thick end, which was pressed into the soft clay to form the inner side of the crucible bottom, as noticeable in the imprints left inside some of them. This is understandable as their size is so small that it would not be manageable to throw them on the wheel or form them around one's thumb.

Finally, there is one crucible (OB 316) that shows a different stamp. In contrast to the simple 'T' stamp described above, the fineness of this stamp indicates that it was made by imprinting a metallic tool. It consists of the letters IE above a stylised wave and a triangular design, all framed by two concentric circumferences (fig. 34). Von Osten (1998: 44, after pers. comm. from W. Endres) suggested that IE could be the initials of Jan Emens, a renowned pottery master who worked in Raeren (Belgium) in the second half of the 16th century (cf. Gaimster 1997: 225). Unfortunately, as it was a unique item on display at the *Museum Der Alchemist*, this crucible could not be sampled for analysis.

Figure 34. Left, typical 'T' stamp on the base of a crucible (diameter of stamp is ~1 cm). Centre, base of big crucible, showing 2 stamps. Right, base of the only crucible with 'IE' stamp (diameter of stamp is ~1 cm).

The colours of the crucibles in the assemblage range from bright white through shades of grey to black, also including orange, pink, red, purple and brown, either as solid colours or in patches. A similarly wide variability exists with regard to the degree of vitrification and density of the fabrics, and surface smoothness. A preliminary optical approach to the crucible samples submitted for scientific analysis did not allow a determination of the number of fabric types represented, given this variability. Given the surface contamination of most artefacts, XRF analyses were not very useful to this end. It was only after microstructural and SEM-EDS chemical analysis of the first samples, and when the entire assemblage was visually examined, that it seemed possible that just one, or very few, clay sources had been used for all the crucibles, their differing appearances being a result of differing temperature, redox and contamination conditions during use, as well as post-depositional processes (figs. 35-36). This observation stresses the limitation of the external appearance of these or other crucible fabrics as a criterion for their classification. In addition, from this starting point, it was possible to compare the condition of unused and used fabrics as a useful methodology to assess their technical standard and performance under various working conditions.

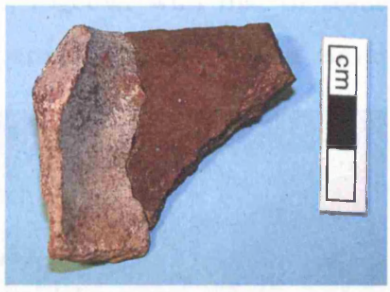


Figure 35. Two matching crucible sherds of different colours, showing the extent of varying post-depositional processes within the same archaeological deposit.

Figure 36. Some crucibles of identical size and fabric, showing different colours as a result of varying conditions of use and post-depositional environments.

With the exception of the smallest crucibles, which are almost invariably white, the standard unused crucible is dark grey or black on the surfaces, although often showing lighter patches. The internal surface is sometimes lighter as well, the same colour as the main body fabric (fig. 37). In all cases, the crucibles show a remarkable surface smoothness. When compared to other artefacts within this and other

assemblages, the 'touch' of the Oberstockstall crucibles, together with that of some other black wares and distillation equipment, appears particularly even. Upon closer examination, some streaks could be noticed on the surfaces, resembling 'brush strokes', sometimes ~2 cm wide, as if made with a brush or cloth (fig. 38). These marks are particularly conspicuous in those crucibles not containing graphite (see below). In view of this, it could be suggested that the crucibles were dipped in liquid, well-refined clay, which was then wiped onto their surfaces, thus making the crucible surfaces as smooth as possible. This outer 'slip', being so thin and well bonded with the body, and sharing the same chemical composition, could not be identified during the SEM examination of cross-sections. Perhaps no clay was added at all, but the potter simply sprinkled some water or used a damp cloth, making their surfaces soft enough to allow this smoothening tamping or wiping. It is worth noting that, in many cases, the fabric of the crucibles seems to have decayed 'in layers', thus giving the impression that they might have been coated with a distinct clay slip (fig. 39). Microscopic and chemical analyses of cross-sections, however, do not support this interpretation.

Figure 37. Three unused crucibles from Oberstockstall.

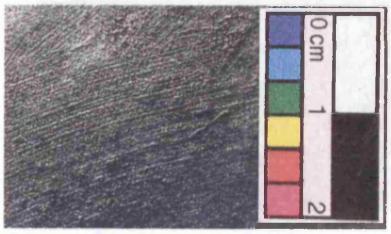


Figure 38. Detail of the surface smoothening of a crucible.

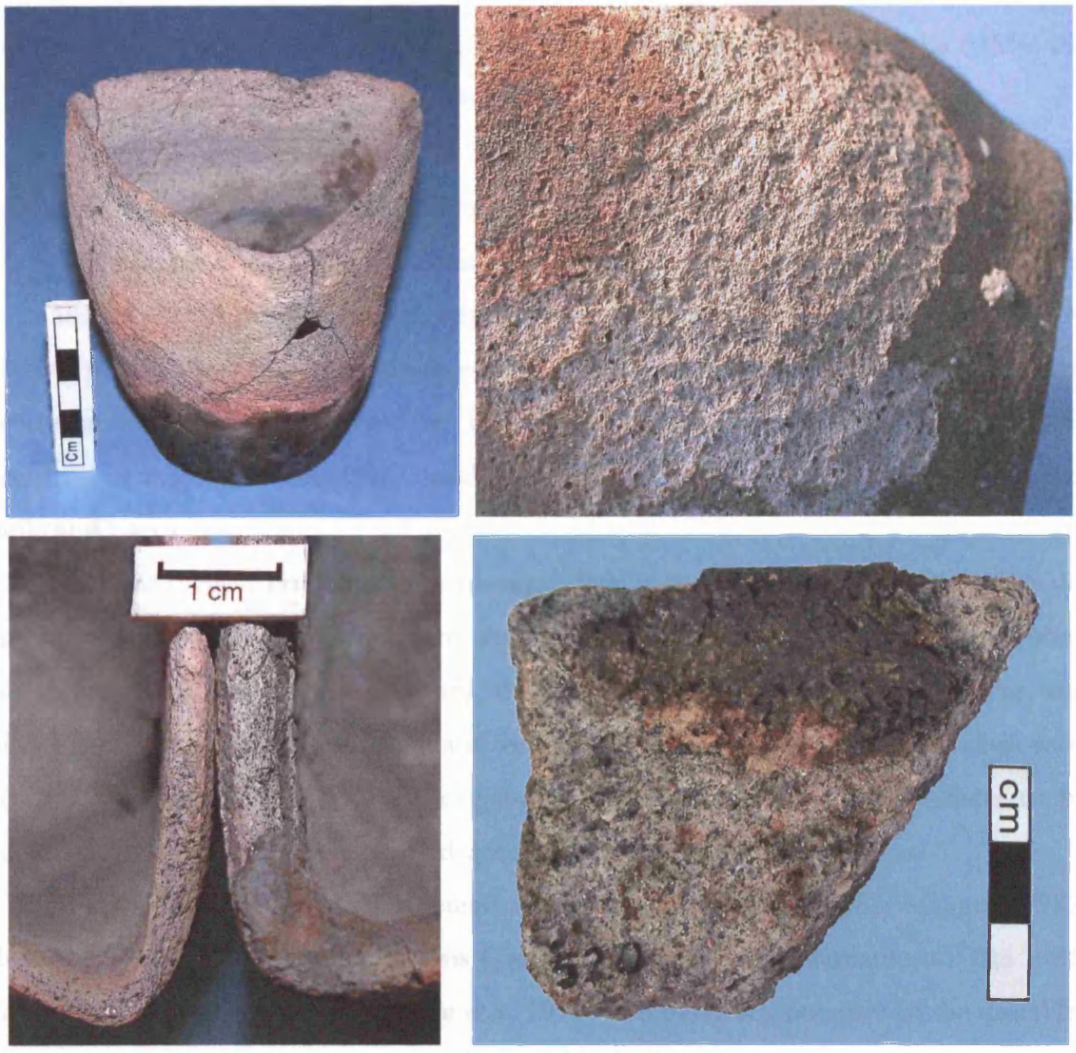


Figure 39. Images of different crucibles showing what might look as remains of a surface slip, but is likely to be just the decay of the more vitrified surface, which comes off the ceramic body.

The ceramic matrix as seen in unused crucibles is fine-grained and shows an advanced stage of initial vitrification, with amalgamated grain boundaries (fig. 40) (cf. terminology in Maniatis and Tite 1981). The most notable inclusions are normally graphite fragments, with speckles up to 0.8 mm long, constituting 20-25 vol% of the fabric (figs. 41-42). The bigger fragments appear oriented with their long axis parallel to the surfaces of the body, especially in the bigger vessels, where this orientation would be easily achieved as a result of their throwing on the wheel. Interestingly, in some of the unused vessels, part of the graphite inclusions have burned off, down to 1 mm below the surfaces – their previous existence noticeable in the still existing elongate voids left behind (fig. 43). This suggests that the original firing in the potter's kiln might not always have been reducing enough so as to prevent oxidation of the graphite. The

fragments of graphite are sometimes intergrown with minerals such as feldspars or iron silicates (figs. 44-45). It should be noted, however, that a few of the crucibles (~5%) did not contain graphite at all (fig. 49; see below).

Besides the graphite, crucible fabrics contain 20-25 vol% of other minerals as inclusions (figs. 40-49). These are subangular and show a moderate degree of sphericity. With the exception of some inclusions up to 1 mm large, the diameter of these is normally below 0.3 mm. Approximately 90% of these minerals are quartz and sodium-bearing potassium feldspar, in about equal proportions. In the non-graphitic crucibles, quartz inclusions are more abundant. The rest are grains of amphibole, iron oxide, rare occurrences of plagioclase, apatite and mica, as well as minute inclusions of ilmenite, rutile and zircon.

In the biggest crucibles, even though their matrix composition falls within the same compositional range, the size and abundance of both silicate and graphite inclusions are higher. On the contrary, the smallest vessels do not have graphite, and silicate inclusions in them are scarce and very small. This suggests that similar clays were processed differently, possibly by levigation, depending on the final products to be manufactured – an indication of the degree of specialisation of these wares.

Based on established parameters of mineralogy (Heimann 1982; Maggetti 1982; Heimann 1989) and SEM observations (Tite and Maniatis 1975; Maniatis and Tite 1981; Tite *et al.* 1982a; Tite *et al.* 1982b; Tite *et al.* 1990), the firing temperature of the crucibles is estimated to have been between 950 °C and 1050 °C. In a recent experimental study conducted with a non-calcareous illitic clay tempered with 25 vol% of quartz-rich sand, Wolf (2002) recorded that plagioclase starts to decompose above 900 °C and potassium feldspar is stable up to about 1050 °C. The presence of residual plagioclase and the unaltered quality of feldspar inclusions in the unused Oberstockstall crucibles indicates a firing temperature between those thresholds. Considering the especially refractory quality of the paste (see below), the moderate degree of vitrification of the matrix further supports this firing temperature estimate.

At first sight, the chemical compositions of the ceramic matrices show a relatively wide variability, especially pronounced in the sodium, sulphur, calcium and chlorine concentrations, but also noticeable in potassium, iron and phosphorus (Table 1). However, the possibility of fabric contamination through use and post-depositional alteration should be considered: in particular, the positive correlation between sodium and chlorine (OB 307, OB 494, OB 515, OB 520) suggests contamination by rocksalt

(NaCl), perhaps used as a flux in the crucibles, whilst that between sulphur and calcium (OB 286, OB 569) may indicate the absorption of gypsum ($CaSO_4 \cdot 2H_2O$) from the burial environment. Unused crucibles without evidence of strong post-depositional alteration show a much narrower compositional range (Table 2). A common ceramic paste source may therefore be assumed for all the crucibles. The slight variations may be attributed to local geological variability as well as to the variable strategies of clay processing noted above (cf. Blackman 1992; Day *et al.* 1999; Buxeda i Garrigós *et al.* 2003), although the possibility of some post-depositional alteration should not be ruled out – significantly, the values showing a higher coefficient of variation in the unused vessels are those for sodium, phosphorus, potassium and calcium, which are some of the most likely elements to be affected by post-depositional processes (Freestone 2001; Buxeda i Garrigós *et al.* 2001; Schwedt *et al.* 2004).

The average composition of the ceramic matrix as detected in the unused crucibles (Table 2) indicates a substantial refractoriness. The alumina concentration is above 30 wt%, with the sum of alkali and earth alkali oxides below 4 wt%. There is, however, a relatively high iron oxide content, reaching 5 wt% in average, whose influence on the crucibles' performance will be discussed below.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO	ZnO	Sb_2O_3	PbO
OB 286	0.5	1.1	28.5	55.6	0.2	3.0	-	2.3	3.0	1.4	4.5	-	-	-
OB 288	0.1	0.6	33.1	58.7	0.4	-	-	1.5	0.6	1.1	3.9	-	-	-
OB 290	0.2	0.7	32.2	57.5	0.2	-	-	1.9	0.6	1.2	5.3	-	-	-
OB 307*	2.5	0.8	31.2	55.0	0.2	-	0.7	5.5	0.6	1.2	2.4	-	-	-
OB 345*	0.4	0.5	30.4	57.4	0.3	3.3	-	2.1	0.6	1.3	3.6	-	-	-
OB 394	0.1	0.8	30.9	58.5	0.2	-	-	2.3	0.6	1.3	5.3	-	-	-
OB 395	0.3	0.7	33.4	56.2	0.3	-	0.2	2.1	0.6	1.3	5.1	-	-	-
OB 466*	0.5	0.5	32.0	56.3	0.2	0.2	≤0.1	2.5	0.7	1.4	5.6	-	-	-
OB 494*	2.5	1.2	29.3	55.4	0.3	-	1.2	7.2	0.6	1.2	1.3	-	-	-
OB 495*	0.2	0.4	32.7	53.9	0.4	-	-	1.8	0.6	1.4	7.9	-	-	0.7
OB 515*	0.5	0.7	34.0	57.3	0.5	0.2	0.2	2.5	0.5	1.4	2.5	-	-	-
OB 519F	0.3	0.7	33.9	56.2	0.4	-	-	2.0	1.0	1.1	4.5	-	-	-
OB 520*	1.9	0.7	32.8	54.7	0.4	-	0.8	2.8	0.4	1.3	4.0	-	-	-
OB 560	0.2	0.7	30.8	56.9	0.2	-	-	2.8	0.8	1.6	6.1	-	-	-
OB 569	0.3	0.5	34.4	56.0	0.3	0.6	≤0.1	1.5	1.6	1.5	3.3	-	-	-
OB n001*	0.4	0.8	30.1	55.1	0.2	-	-	2.5	1.2	1.5	7.1	0.2	1.0	-
Std. dev.	0.82	0.21	1.76	1.35	0.1	1.55	0.43	1.51	0.64	0.14	1.74	-	-	-
Coeff. var.	120	29.5	5.5	2.4	34.0	106.0	69.4	55.8	73.1	10.6	38.5	-	-	-

Table 1. Average composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of different crucibles from Oberstockstall, with all detected elements. The star (*) denotes crucibles with evident traces of high-temperature use. The bottom rows show the standard deviation (σ) and the coefficient of variation (%) for each element. See Appendix 1 for detailed analytical results.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
Mean (n=6)	0.2	0.7	32.4	57.3	0.3	2.1	0.7	1.3	5.0
Std. dev.	0.08	0.08	1.32	1.10	0.08	0.43	0.16	0.19	0.74
Coeff. var.	40.0	11.4	4.1	1.9	26.7	20.5	22.9	14.6	14.8
Max.	0.3	0.8	33.9	58.7	0.4	2.8	1.0	1.6	6.1
Min.	0.1	0.6	30.8	56.2	0.2	1.5	0.6	1.1	3.9

Table 2. Average composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of unused crucibles without evidence of strong post-depositional alteration (OB 288, OB 290, OB 394, OB 395, OB 519F, OB 560). The bottom rows show the standard deviation (σ), the coefficient of variation (%), and maximum and minimum values (wt%). For compositions of individual crucibles, see Table 1 and Appendix 1.

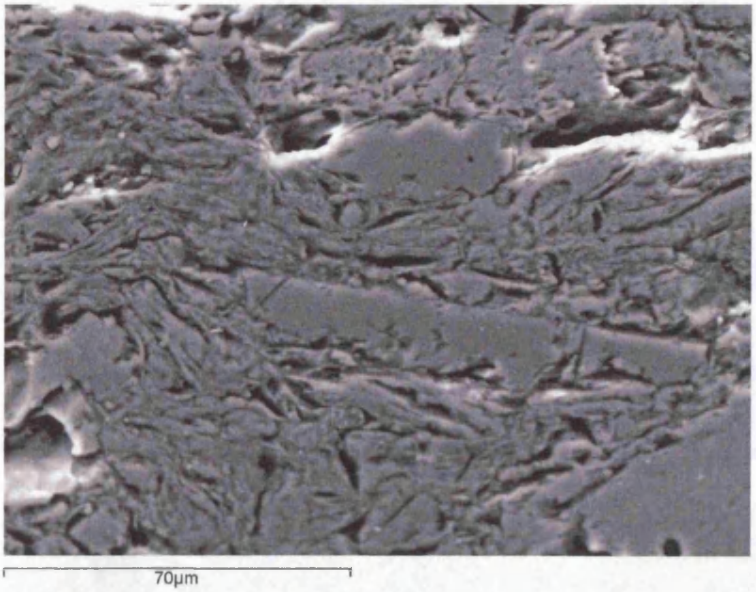


Figure 40. SE photomicrograph of an unused crucible at high magnification, showing an advanced stage of initial vitrification: clay platelets are buckling but the layered structure is still noticeable (OB 395/s1, 800x).

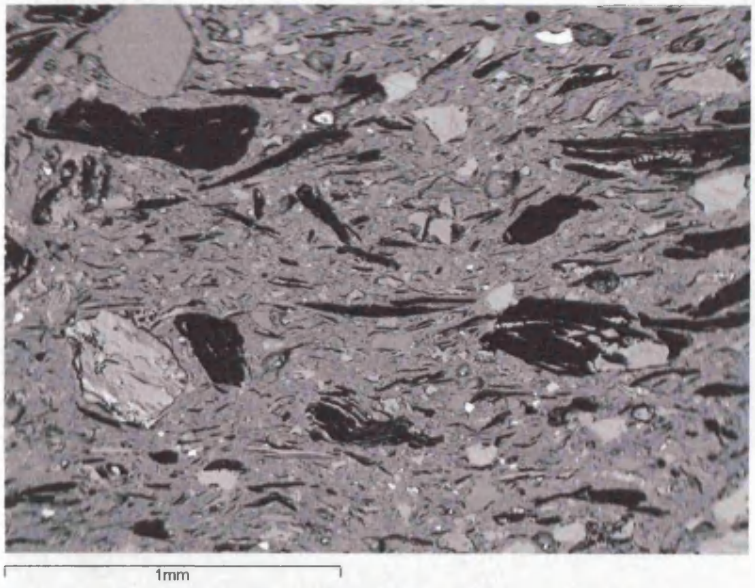


Figure 41. BSE photomicrograph of a crucible at low magnification. The black elongate particles are graphite inclusions; dark grey inclusions are quartz, light grey ones are feldspar, white ones are zircon, ilmenite and rutile (OB 290/s1, 50x).

Figure 42. Photomicrograph of a crucible showing abundant graphite and silicate inclusions (OB 519F/s1, XPL, 50x, long axis represents ~2 mm).

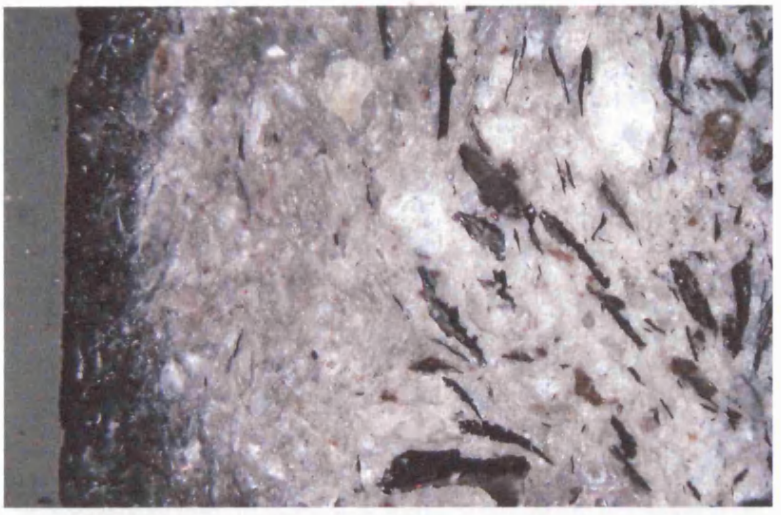


Figure 43. Photomicrograph of a cross-section including the outer surface of an unused crucible (left), where the graphite inclusions have burned off during firing. Note, however, the black smoking of the surface, discussed later (OB 519F/s1, XPL, 50x, long axis represents ~2 mm).

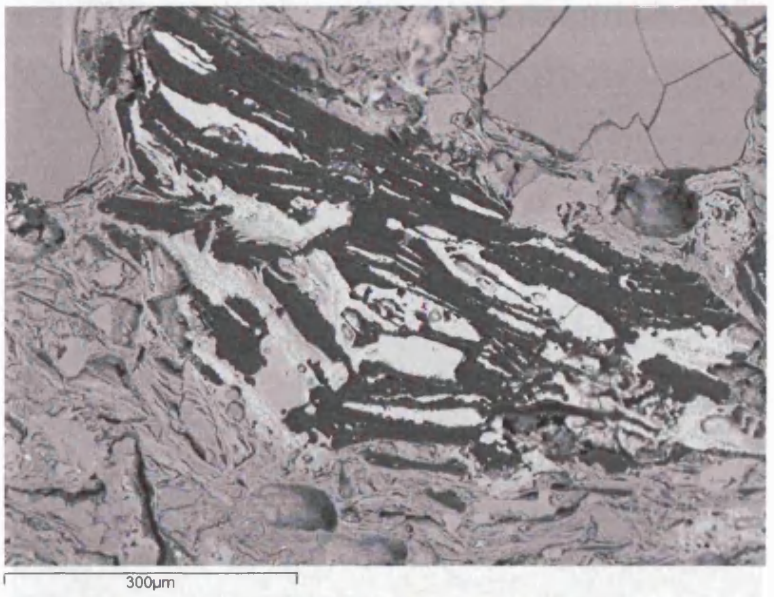


Figure 44. Graphite inclusions intergrown with an iron silicate that is decomposing as a result of the high temperatures (OB 395/s1, BSE, 150x).

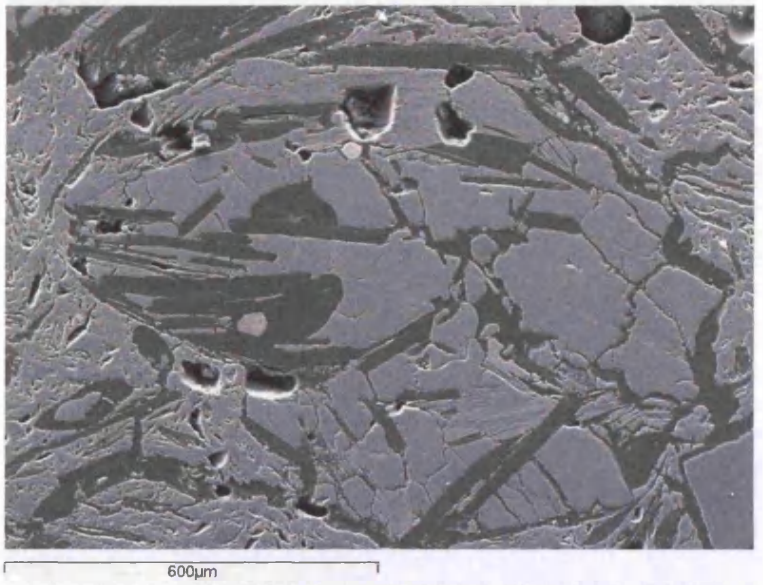


Figure 45. Another graphite inclusion intergrown with silicates. The core is feldspar, the brighter phases being zircon and iron silicate (OB 520/s1, SE, 100x).

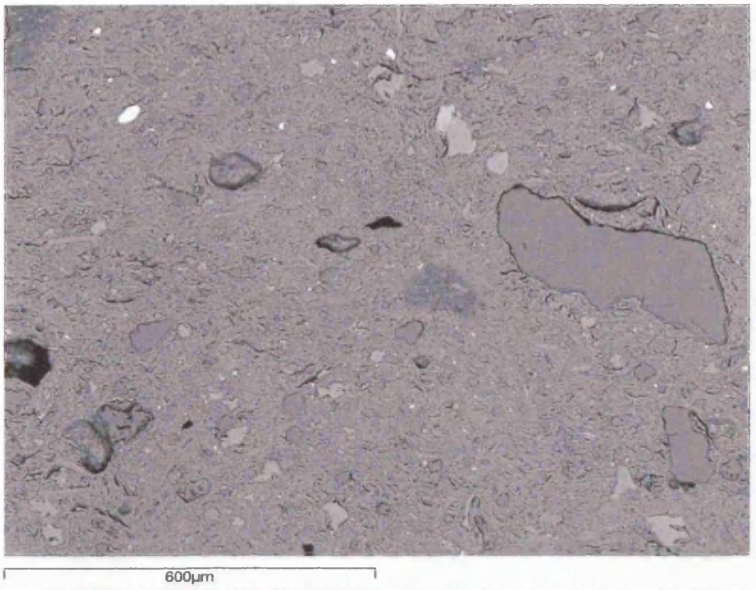


Figure 46. BSE photomicrograph of the ceramic fabric of one of the smallest crucibles, showing the absence of graphite, and the scarcity and generally small size of silicate inclusions (OB 569/s1, 100x).

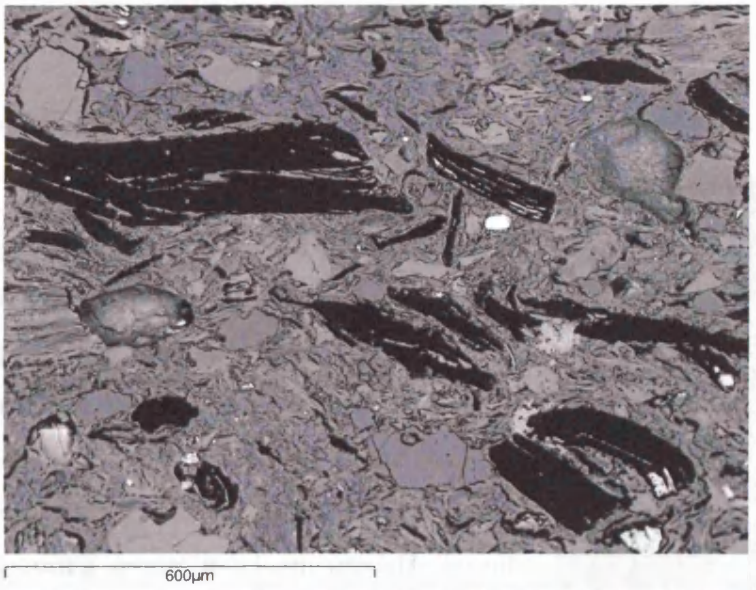


Figure 47. BSE photomicrograph of a bigger crucible to the same scale, showing the presence of graphite as well as the higher concentration of silicate inclusions (OB 395/s1, 100x).

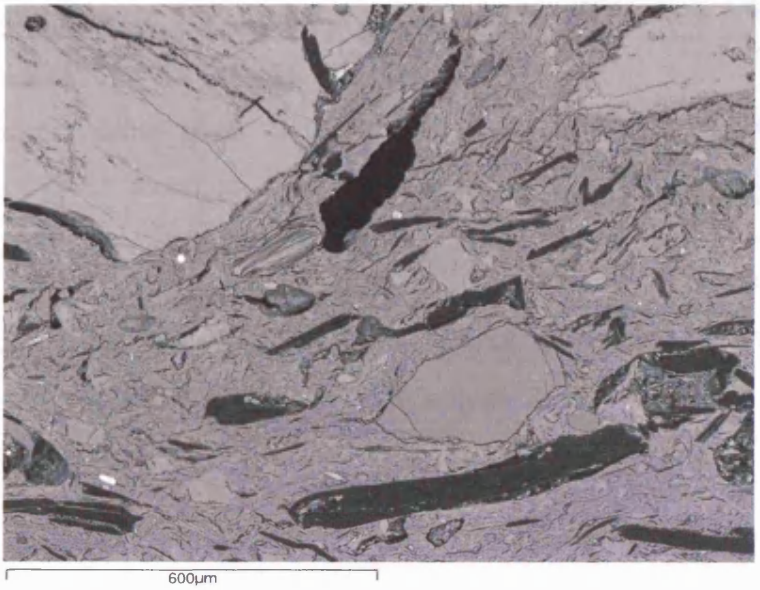


Figure 48. BSE photomicrograph of one of the biggest crucibles to the same scale, with even bigger inclusions. Note that the biggest ones were not included in the picture because they covered the whole field of view (see fig. 45) (OB 520/s1, 100x).

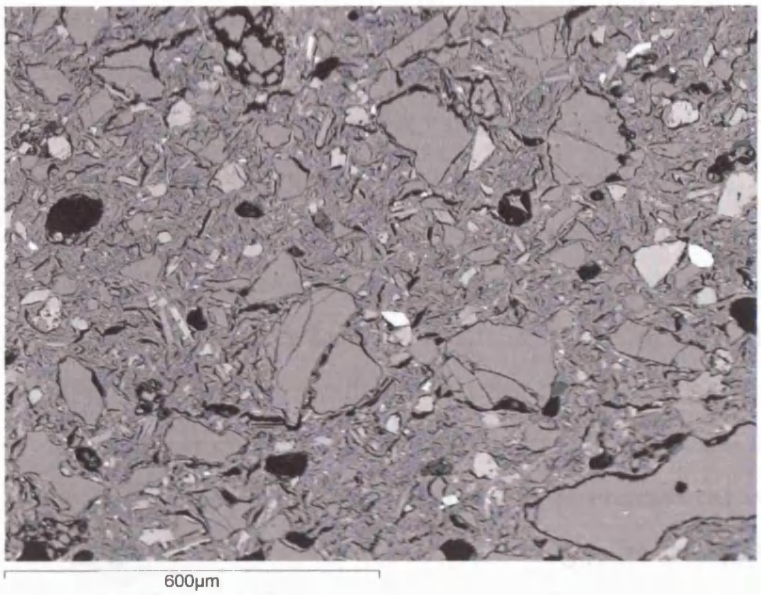


Figure 49. BSE photo-micrograph of one of the few non-graphitic scorifiers in the assemblage, showing a slightly higher concentration of quartz inclusions (dark grey). Compare to fig. 47 (OB 394/s1, 100x).

6.2.2. Discussion

Starting with the formal properties of the vessels, the triangular mouth was an easy technical solution providing three convenient spouts which allowed handling and pouring with tongs from any direction. In some of the crucibles, it can be seen that one of the pouring spouts has been used to decant slag, and another one for pouring molten metal. The flat base and the absence of direct firing traces indicate that these crucibles stood in a chamber separate from the fire box within the assay furnace. This indirect heating mode prevented direct contact between the fuel and the vessel, thus improving stability

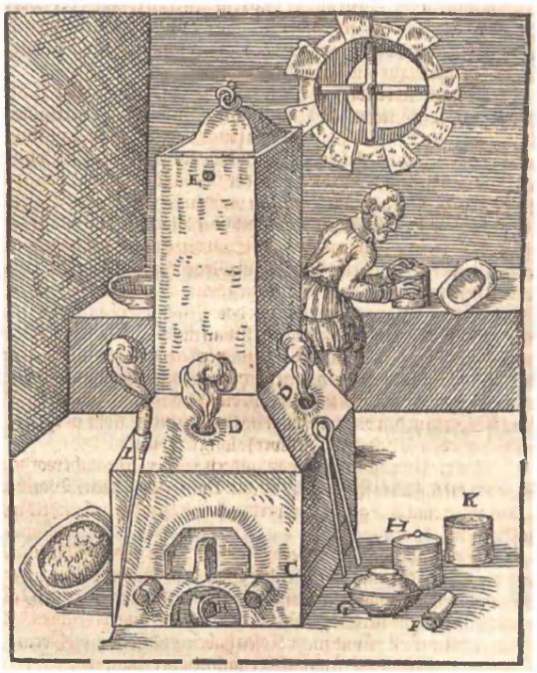


Figure 50. Example of a 16th-century assay furnace, showing the chamber separated from the fire box by a flat platform (marked with a 'C') (from Sisco and Smith 1951: 185, plate 27).

and control whilst minimising contamination of the crucible charge and corrosion by fuel ash (fig. 50)[14]. These flat-bottomed crucibles, easier to make in large numbers on a potter's wheel, differ from earlier vessels that were to sit within the uneven charcoal bed and, accordingly, were designed with concave bases (Bayley 1996).

The different sizes of the crucibles were most likely related to the volume of the charge to be processed in them. The body thickness was probably reflecting the expected weights of the loads. A relatively thick crucible wall would also have provided a buffer for corrosion by fluxes and hot metal oxides, and indeed some of the used crucibles appear corroded to varying depths. However, too thick a crucible would make heat transfer much slower, as well as it would increase the risk of failure due to a steep thermal gradient through the wall. Accordingly, we might infer that the ideal thickness for a crucible would be a compromise: as thin as possible, but within the limits set by the required physical strength and stability against corrosion. It should be noted that the crucibles are thicker than most of the other ceramic vessels in the assemblage, especially those vessels apparently not designed for technical use.

The relatively closed shape of the vessels would facilitate the control of redox conditions. Reactions carried out in the crucibles would often require a reducing atmosphere, for example the fluxing of ores resulting in the primary concentration of the metals (see below, section 6.2.4). The Oberstockstall crucibles appear sometimes lidded with lute, a recycled sherd, or an upturned scorifier, which would further limit the presence of oxygen. Possibly, these crucibles were used shut quite frequently, in particular with upturned scorifiers as lids. This would explain one of the two symbols used to represent crucibles in the 18th-century *Encyclopédie*, which the remains from Oberstockstall help interpret (fig. 51). Often the ceramic wall of the crucibles shows an orange or brown colour on the outer surface, and becomes grey towards the inside, showing that although the atmosphere in the furnace chamber was oxidising, the reaction inside the vessels took place in reducing conditions.

[14] If operations required a more direct heating, the vessels could be protected by being placed under a muffle.

Figure 51. Several ways of lidding a crucible documented in Oberstockstall, all of them showing recycling and the versatility of the tools. Clockwise from top left: a) a luted sherd; b) an upturned crucible bottom, luted; c) an upturned scorifier (left, as found; right, as reconstructed; inset, symbols representing crucibles in the *Encyclopédie*); d) a lump of lute, which was possibly used in combination with something else.

Turning to the material properties of the ceramic fabric, it was noted above that the chemical composition of the matrices indicates the use of a thermally rather refractory clay, a very expedient quality given that the vessels would often be used at temperatures reaching or exceeding 1000 °C (see below, section 6.2.4). The question arises whether this indicates a deliberate choice of the most suitable clays for the crucibles, or whether refractoriness was a common quality of the bulk of the clay ordinarily used by the potters. When comparing the chemical composition of these vessels to those of other artefacts from the laboratory, and to ordinary wares found at the same site and others in the region, the ceramic of these vessels ranks amongst the most refractory ones (see sections 6.5 and 7.5). However, it should be noted that other artefacts that would not be subjected to such high temperatures, including ceramic containers, were also made of rather refractory ceramics. Thus it seems that the availability of refractory clays was not a critical issue, and that 'better' clays were routinely identified and used, rather than keeping them for the thermally most demanding technical ceramics.

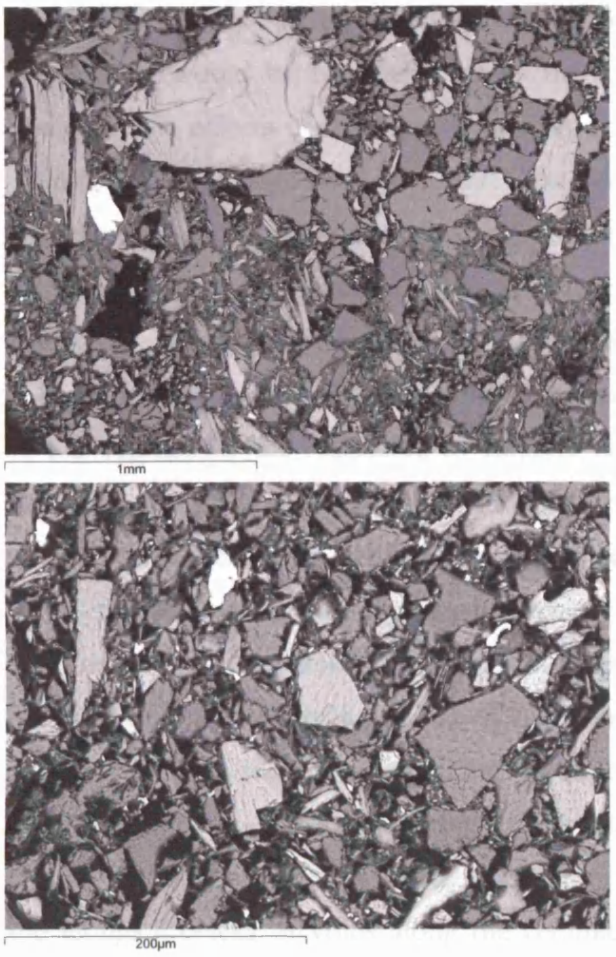


Figure 52. Two views of the clay sample from Obernzell at different magnification, showing the remarkable angularity of the silicate inclusions (OBZL clay/s1, BSE, top at 50x, bottom at 300x).

A further effect of the chemical composition of the alumina-rich ceramic matrix may have been thermal conductivity, and this would be beneficial for most of the artefacts analysed – whether crucibles, cucurbits or cooking pots. Recent analyses of modern refractory ceramics (Akiyoshi *et al.* 2003) have demonstrated that there is a positive correlation between alumina content and thermal conductivity. However, this study was based on ceramics with alumina concentrations at or above 35 wt%, hence we have no real data extrapolative to the present case.

It was not easy to discern whether the silicate minerals within the matrix were a natural component of the original clay, or whether they were artificially added by the crucible makers as temper. At first sight, the shape of these inclusions appeared rather angular, especially when compared to the clearly spheroid sand in other contemporary crucibles (see chapters 8 and 9), which led the author to hypothesise that they could constitute crushed minerals mixed with the clay. However, similarly angular inclusions were identified upon examination of a clay sample from Obernzell, in the region where these crucibles were originally thought to have been made (see chapter 7), in proportions even higher than those documented in the crucibles, hence it seems feasible that these were present in the original clay (fig. 52). Furthermore, the relative abundance of different inclusion sizes did not unambiguously suggest the bimodal distribution typical of artificial temper. However, it should be noted that the few non-graphitic crucibles contained more quartz inclusions, in spite of the similar matrix composition (see discussion below).

Whether natural or artificially added, the presence of angular inclusions within the ceramic would be beneficial for its mechanical properties. Recent experimental studies of the effects of different temper types on the material properties of ceramics (Kilikoglou *et al.* 1998; Tite *et al.* 2001) have demonstrated that the presence of non-plastic inclusions helps arrest the propagation of cracks developed upon thermal shock, particularly when they have an angular shape, as these inclusions stop and deflect these cracks and require more energy for them to catastrophically expand through the ceramic bodies. For the same reason, this type of inclusions would also enhance the toughness of the vessels, *i.e.* the resistance to fracture due to sudden impacts, for instance as caused by accidental mishandling or when deliberately striking the crucible for the liquid metal to collect in the bottom (e.g. Smith and Gnudi 1990: 140).

Similar arguments had been put forward by Ann Woods (1986), and taken up by other researchers (Freestone and Tite 1986; Freestone 1989; Bayley *et al.* 1991: 402) after the SEM examinations of a number of crucibles. Focussing on quartz inclusions, these authors explained that this mineral suffers a notable increase in volume when heated through 573 °C in the original firing, in the lattice inversion of α- to β-quartz. Upon cooling and contraction, the quartz grains leave characteristic voids around themselves and elongate cracks or voids along the ceramic body. Subsequently, during the high-temperature use of the vessel, these voids will accommodate the expansion of the quartz. In this sense, quartz grains could function as a "dimensional stabilizer" (Freestone 1989: 159). In the Oberstockstall crucibles, however, contrary to others (see references above and chapter 9), the characteristic elongate voids are not particularly noticeable, perhaps because the flexible graphite inclusions arrest the propagation of cracks.

Even if unintended, this relatively low porosity of the fabric would probably be beneficial for the crucibles' performance. Firstly, since air is not a good conductor of heat, the lack of voids would enhance the thermal conductivity of the fabric and decrease the thermal gradient through the crucible wall during initial stages of firing during use (cf. Schiffer and Skibo 1997: 605; Sillar 2003). Secondly, a denser matrix would help prevent the penetration of fluxes and corrosive metal oxides, as well as prills of molten metal from being trapped within the fabric. This would be particularly relevant if quantifiable fire assays were conducted.

The most special feature of most of these crucibles is their graphite content. The presence of graphite in the ceramic paste would have enhanced most of the technical

parameters of the crucibles. Being one of the most stable minerals under high temperatures – it does not melt but only sublimates at *ca.* 3500 °C –, graphite would have contributed to the vessels' thermal refractoriness. Although graphite is just an inclusion within the paste, thus not affecting the heat resistance of the matrix itself, the rather high proportion of graphite inclusions would probably enhance the refractoriness of the fabric altogether. Furthermore, due to its chemical inertness, these crucibles would remain significantly less attacked by the potentially corrosive charge and the forming slag. If reacting with the charge – as seen in some of the used vessels (see below) –, it would also be favourably, in this case to inhibit oxidation or favour reduction, since graphite is pure carbon. Although the mechanical behaviour of graphite as ceramic temper remains uninvestigated, we may assume that its platy shape and flaky fracture, together with the toughness of graphite speckles in the long axis, could make it ideal for preventing the propagation of potentially fatal cracks across the body of the fabric, as is the case of mica (Tite *et al.* 2001), which has a very similar crystallographic structure. This would very significantly increase both the toughness and the thermal shock resistance of these crucibles. The potentially catastrophic expansion and contraction of ceramics with changing temperatures would also be significantly lower in graphitic fabrics (Duma and Ravasz 1976). Furthermore, given the flexibility of graphite flakes, the presence of these inclusions may have enhanced the tensile strength of the vessels (*i.e.* the resistance to fracture due to sustained pressure), which tends to be the main weakness of ceramics heavily tempered with aplastics (Kilikoglou *et al.* 1998; Tite *et al.* 2001)[15]. Since these crucibles often had to be quickly removed from the furnace for pouring while hot and holding considerably heavy loads, these mechanical properties would be very advantageous. Graphitic crucibles are traditionally reported as extremely resistant to thermal shock and capable to withstand repeated runs (Percy 1875: 122). Finally, graphite being an excellent heat conductor, it would improve the thermal conductivity of the vessel, hence allowing steeper heating rates and savings on time and

[15] Laboratory measurements of the material properties of graphite-tempered ceramics are eagerly expected (but see Duma and Ravasz 1976). One aspect deserving specific attention is the tensile strength of these vessels. Studies so far suggest that, in most tempered ceramics, there is a negative correlation between, on the one hand, thermal shock resistance and toughness, and, on the other hand, tensile strength. It is suspected that graphite might constitute an exception, improving all three properties. In the measurement of the tensile strength, it would be necessary to consider whether the crucibles were manipulated either by 'pinching' the rim or by embracing the whole body with tongs, as well as different charge weights and temperatures. Finite element analyses could provide a useful methodology to assess this (cf. Kilikoglou and Vekinis 2002).

fuel. The counterpoint of this aspect would be the quicker cooling rate of the crucibles, if the content had to be poured.

Two important questions emerge. Firstly, was graphite naturally present in the clay, or was it deliberately added? Secondly, were potters and crucible users aware of the effects of graphite inclusions in the material properties of the fabrics?

At first sight, it is tempting to believe that graphite was artificially added to the clay. This idea would be supported by the presence of some crucibles that, whilst showing the same matrix chemical composition (Table 3) and types of silicate inclusions as the others, do no bear any graphite. Given that non-graphitic crucibles show a higher concentration of quartz inclusions (figs. 47 and 49), we could conclude that some crucibles were tempered with graphite, whereas others were tempered with silicates.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
OB 395	0.3	0.7	33.4	56.2	0.3	2.1	0.6	1.3	5.1
OB 519F	0.3	0.7	33.9	56.2	0.4	2.0	1.0	1.1	4.5
OB 290	0.2	0.7	32.2	57.5	0.2	1.9	0.6	1.2	5.3
OB 288	0.1	0.6	33.1	58.7	0.4	1.5	0.6	1.1	3.9
OB 560	0.2	0.7	30.8	56.9	0.2	2.8	0.8	1.6	6.1
OB 394	0.1	0.8	30.9	58.5	0.2	2.3	0.6	1.3	5.3

Table 3. Average composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of different crucibles. The four top rows represent graphitic crucibles, whereas the bottom ones are made of non-graphitic fabrics. The slight differences in alumina and potash may be related to varying abundance of potassium feldspar, but all the crucibles may reasonably be included within the same compositional group.

There are, nevertheless, archaeological and historical reasons to argue against this supposition. First of all, none of the numerous 16th-century and earlier written sources reviewed mention the addition of graphite to clay as temper – not even do they refer to graphite as such at all. This is surprising if we bear in mind, for example, Biringuccio's, Agricola's and Ercker's familiarity with Central European chymistry and metallurgy, the references to 'Vienna' crucibles made in the *Probierbüchlein* (Sisco and Smith 1949: 110) and *De la pirotechnia* (Smith and Gnudi 1990: 72) [16], and their explicit and detailed descriptions of other temper types for crucible making, such as sand, grog or crushed fired bricks (see chapter 10.2). We know, in contrast to this, the appreciation

[16] Cotter (1992: 266) refers to these as "graphitic Vienna crucibles", although there is as yet no evidence to prove that these vessels were graphitic, or indeed equalling those used in Oberstockstall, some 40 km from Vienna.

shown by Bavarian potters – producers of crucibles and other black wares – , to *'Eisentachen'* ('iron clay'), a term that may well originate from the dark, shiny lustre of the existing naturally-bearing graphite clay deposits (Bauer 1983 and section 7.2). If graphite was deliberately added to the clays, we could presume that potters did so because they appreciated some of the resulting technical advantages. However, following this line of reasoning, then one could ask why they left some crucibles untempered.

This leads us to the second question: whether naturally or artificially graphitic, were these clays particularly valued as the most suitable for technical ceramics? Within the analysed sample of the Oberstockstall assemblage, and in comparative archaeological ceramics from nearby Obernzell, high-graphite ceramics appear invariably used for crucibles and scorifiers (see section 6.5 and chapter 7). This could be taken as an indication that the most suitable clays were identified and reserved for the thermally, chemically and mechanically most demanding wares. The interpretation is not so straightforward, though. On the one hand, graphitic ceramics have been recorded in medieval and early modern domestic pottery from other Austrian sites (Duma and Ravasz 1976; Hebert and Lehner 1995), and seen by the author in the *Keramikmuseum* in Obernzell. On the other hand, as noted above, some of the crucibles are not graphitic.

This combination of factors suggests that naturally graphitic clays were selected – probably identified by their appearance –, but there are no reasons to assume that 16th-century potters had yet made the link between these clays and the mineral graphite. By experience, and necessarily through feedback between manufacturers and end users, it would be realised that those crucibles made of 'dark clays' performed better than others, which might have led to progressively reserve graphitic clays for technical ceramics. This may have been a relatively long process, not noticed at once, which would explain why, in spite of this trend, there is no clear-cut division between technical/graphitic and ordinary/non-graphitic ceramics. This hypothesis will be further discussed in section 7.2.

As regards the surface smoothening of the crucibles, several explanations may be proposed, which are not mutually exclusive. To start with, this smoothness would further enhance the vessels' resistance to chemical attack or metal loss, by reducing the surface area and, hence, the possibility of penetration by the charge (Bayley *et al.* 1991: 404). Another technical advantage of this would be that, upon vitrification, this thin layer of fine clay would form a coating that would hinder the burning away of graphite inclusions reacting with any free oxygen in the furnace chamber. Both of these

hypotheses are suggested by John Percy (1875: 123) in his discussion of graphitic crucibles – the latter would, however, leave this smoothening unexplained for the case of the non-graphitic crucibles. A third possibility comes from the modern crucible-making industry (pers. comm. Paul Encoyand, Budget Casting Supply, 2003). Some modern crucibles come from the factory with a painted-on coating that protects the vessel from moisture absorption during initial storage and shipping. Others contain additives that sinter or fuse on the surface after the first firing to help protect against moisture and metal migration. Crucibles that have absorbed moisture can fail or explode during the first use at temperatures as low as 200 °C, even if heated slowly, possibly as a result of water vapour trapped within the relatively dense fabric.

This is not to say that any of these reasons is necessarily behind the choice of smooth surfaces in the crucibles found in Oberstockstall. In fact, the author is inclined to consider that the main factor leading the potters to make their crucibles smooth was a concern with the external appearance of the vessels, as discussed in the following section.

6.2.3. Black crucibles and blue jeans: appearance matters

Together with the surface smoothness, the most notable external feature of the crucibles is their colour. The colour of a crucible is primarily a function of the fabric composition and the firing conditions, provided that there is no other treatment after firing. The colour can be changed in a relatively short time, hence the surface colour of the ceramic informs only about the conditions of the last stages of their last firing. This is corroborated by some of the used crucibles, whose surfaces appear in shades of red – due to the oxidation of the iron content in the fabric – but where graphite has remained, indicating that the oxidising conditions were not kept for a long period (fig. 53). More significantly, some other crucibles are black, *i.e.* have been reduced fired, but graphite has apparently burned away in an earlier, oxidising stage (fig. 43, p. 115).

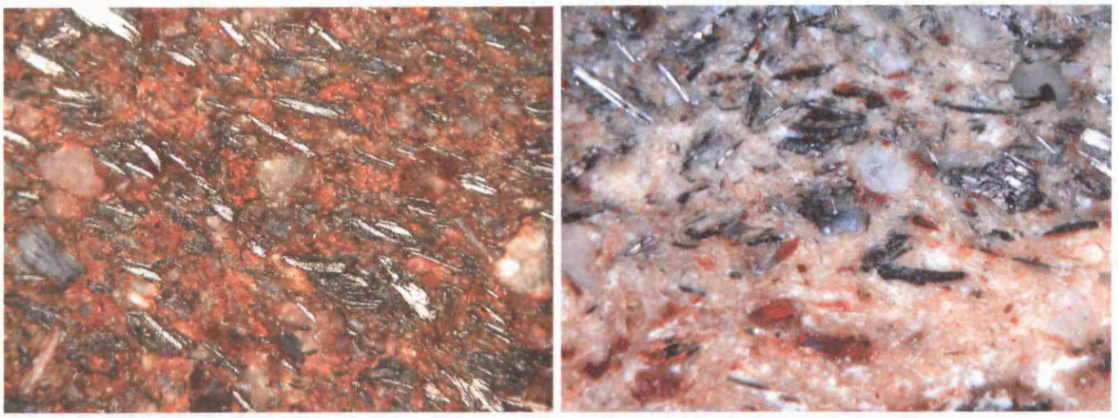


Figure 53. The photomicrograph on the left shows the fabric of a crucible where the oxidising atmosphere resulted in the colour of the paste turning red, but it did not burn away the graphite. In contrast, the photomicrograph on the right shows a crucible where the oxidation starting from the outer surface (bottom of the image) progressively caused the red colour but also the burning away of graphite inclusions (left, OB 515/s1, sXPL; right, OB 495/s1, XPL; both at 50x, long axis represents ~2 mm).

With the exception of the very small, hand moulded vessels, the surface colour of the unused crucibles from Oberstockstall is dark grey or black, although often showing lighter patches (fig. 37, p. 109). This is a result of a smoky firing, possibly the effect of adding grass or any other poorly combusting material to the potter's kiln in order to make an atmosphere rich in hydrocarbons, which would add the blackening soot to the vessels' surface. At early stages of this research, this colour was interpreted as a consequence of having to fire the crucibles in reducing conditions, in order not to burn away the graphite inclusions. The blackness would thus be simply a side-effect of another technical requirement. The fact that a small part of the graphite appears to have burned off in some of the unused crucibles would suggest that the necessary reducing conditions were either not easy to achieve or to control. Experimental firings of reproductions of medieval pottery (e.g. Dawson and Kent 1987) have shown that the control of reducing conditions for decorative purposes is relatively easy. However, with increasing temperatures, the oxidation of graphite is more readily reached (Xiaowei *et al.* 2004), hence preventing the burning away of graphite for prolonged firings might have been more difficult than simply obtaining a black surface colour.

The problem was that the interpretation of the black colour as an unavoidable side-effect of the manufacture did not explain why those crucibles not containing graphite had been blackened as well (fig. 54). Given the similarity in appearance, chemical composition and mineral inclusions (with the exception of graphite) between the two types of crucibles, it seemed acceptable that they had been made in the same region, probably by the same producer. In any case, for these crucibles, the black colour could not be explained as accidental.

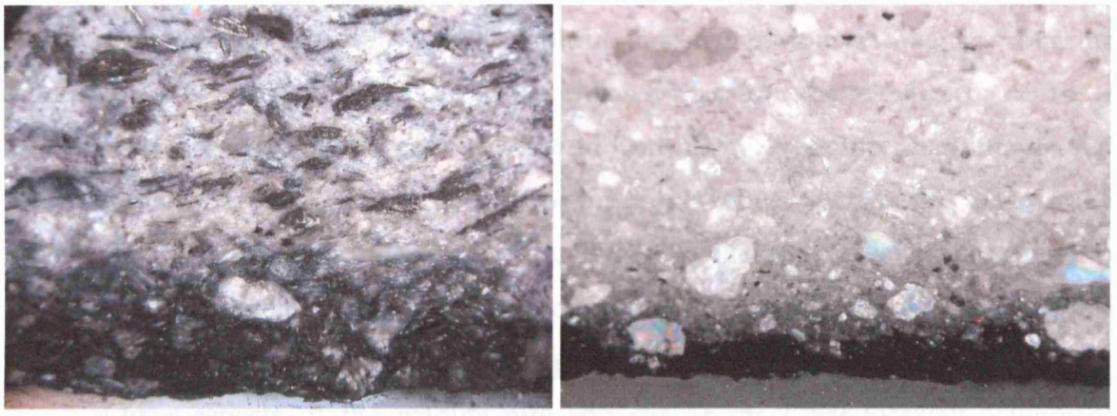


Figure 54. Left, photomicrograph of crucible OB 395 showing graphite inclusions (black phases), silicate inclusions (translucent phases) and the surface blackening (bottom). Right, photomicrograph of crucible OB 560, where there is no graphite but the surface has been blackened (both images XPL, 50x, long axis represents ~2 mm).

If modern commercial concepts are brought up to explain this deliberate blackening of the crucibles, the idea of a potter trying to deceive by mixing some cheaper, non-graphitic crucibles within the stock would appear tempting. Interestingly, an order was issued in 15th-century Vienna whereby it became mandatory that all the black wares in the market should bear maker's stamps, and this may attest the existence of counterfeit imitations of some pottery types (von Molthein 1910: 389). However, if – contrary to the arguments above – potters did clearly distinguish between graphitic and non-graphitic clays, then we should expect the users to be able to recognise them too. As soon as one of these crucibles was broken, the deceit would be uncovered, the word spread, and the supposedly cunning business would quickly become unprofitable.

A more daring interpretation may be raised, which would require abandoning the present-day 'logic' and trying to understand the 16th-century way of thinking that conceived and perceived these crucibles. Our modern understanding of matter tells us that some important technical advantages of these vessels were achieved by using clay containing graphite, a crystallised allotrope of carbon with particular chemical and

mechanical characteristics. That they tend to be black is an accidental, unimportant feature of these crucibles resulting from their graphite content and the firing in reducing conditions. What makes the crucible good is the graphitic 'matter' contained in the fabric, and not the black 'quality'. For a Renaissance person, however, the logic might have been different, and perhaps the black colour could in fact be seen as a technical feature affecting the performance of the crucibles. At this time, the Aristotelian worldview and the corresponding theory of matter were still widely accepted. According to this view, what made things different from each other, what really counted, was not matter – which was one and only, mainly manifested in four elements – but the attributes of matter (see section 1.2). It follows that, for them, what made these crucibles special may have been the black and lustrous quality rather than 'the matter' which made them black. Maybe they were making crucibles that looked alike in the belief that they would perform alike. For us, all the crucibles looked equal and were only discriminated after microscopic examination. For a Renaissance man, they looked equal and, possibly as a result of this, they were equal indeed.

Other aspects of the manufacture and use of the Oberstockstall equipment show, in contrast, a rather modern understanding of the thermochemical behaviour of different materials, probably acquired by experience (see section 6.2.4). In this sense, the importance given to the appearance of the crucibles could illustrate the very intellectual atmosphere of the time, where 'the old' and 'the new' were still coexisting, only starting to clash. The observations carried out during these experiments would lead, in the long run and with much struggle, to the abandonment of the Aristotelian theory of matter, but this step was complex and perhaps in 16th-century Oberstockstall it was not fully accomplished.

The explanation of the black colour of the crucibles in Aristotelian terms was a very useful 'working hypothesis' that made the author fully aware of the need for avoiding present-day categories and assumptions in the interpretation of a society that did not necessarily think in the same way as we do. This vantagepoint is useful and it will be kept as a general premise, but a look at the assemblage from a wider perspective led the author to question the original explanation made in purely Aristotelian terms. This hypothesis is challenged by the fact that some ordinary ceramics of the same region and period, including containers and cooking pots, show a very similar black lustre, even though they would not have been subjected to the thermal, chemical and mechanical stresses usual for the triangular crucibles (see fig. 25 and section 6.5). It can thus no

longer be argued that the black, lustrous quality was seen as a factor specifically enhancing the functioning of high-temperature ceramics. At most, this quality would be seen as 'generally good' for any kind of pottery. Still, it was clear that colour was a choice. Why was care taken in making all the crucibles look alike? Yet another hypothesis may be suggested.

From the perspective of makers and users, pottery has "three directly perceptible characteristics which formed the basis upon which it was conceptualised and classified. These are texture, colour and shape" (Cumberpatch 1997b: 126). Even if other aspects such as the weight of the vessels, their 'ring' when struck with a finger, the price or even the reputation of the merchant could be considered, those three would be the basic factors for a customer to identify one of a kind within a range, and possibly choose it. We can categorise the three of them altogether and, in the case of the crucibles from Oberstockstall, we would have a set of features – smooth, black and triangular – that represented the crucible as such and perhaps stood for a more complex set of qualities. The appearance of these crucibles could thus be a signifier. Modern blue jeans may be used as a suitable example to illustrate this[17].

Denim trousers were usual for workmen in the 1860s. The main innovation and advance took place when Levi Strauss and Jacob Davis designed and patented the use of metal rivets at the points of strain, such as the pocket corners and the base of the button fly, in order to strengthen the trousers. This was the feature patented in 1873 that marks the invention of blue jeans (Downey 2001; Levi Strauss & Co 2001). Other 'technical' assets were double hems or an especially strong fabric from Manchester, New Hampshire. The blue colour was due to the use of the particularly cheap indigo dye, which turned out to be advantageous in that it did not show dirt as much as natural, uncoloured denim. Blue, however, became the signifier by which customers would identify a whole package of qualities – 'blue jeans' were the good, strong ones, readily recognisable by anyone. In the 1930s, Levi Strauss & Co adopted the cowboy as an advertising icon, hence furnishing the blue jeans with a new connotation: the rugged individualism of the cowboy (by this time, particularly in his Hollywood incarnation). Progressively, the other qualities lost preponderance and, by the 1970s, few people would know that the 'essence' of jeans were the metal rivets and other strengths. In the long run, 'blue jeans' were no longer made of denim, they were coloured with synthetic

[17] I am very grateful to Thilo Rehren, who brought up this simile, sparking off my curiosity about the history of blue jeans and its relation to crucibles.

dyes and they did not necessarily have rivets. Nonetheless, blue remained as the condition *sine qua non* for jeans to be conceptualised, classified and demanded. Colour remained a signifier, even if the signified meaning was diluted or even had changed – now, to denote 'fashion'. Only recently have people started to produce and buy jeans in other colours, and their use still is a fashion statement: some customers choose alternative colours precisely because they are *not* blue, which emphasises that colour still matters.

What does this have to do with the Oberstockstall crucibles? The signifying attributes 'triangular, smooth and black' – like 'jeans, riveted and blue' – seem to stand for a more convoluted set of qualities. Graphitic or not, black crucibles could be perceived as the ones from a particular supplier, maybe the same that had a reputation for producing black wares for centuries (see chapter 7). Although, in practice, their performance would differ as a result of different inclusions within the fabric, their material properties could be expected to be generally acceptable. They were 'the' black, smooth, triangular crucibles; quite possibly, 'the well-known' black, smooth, triangular crucibles that a consumer would look for in the market or in the laboratory stock, whichever the reasons for this in any given context.

Studying patterns of pottery production and consumption in present-day Andean communities, Bill Sillar (1997; 2000b) has shown the crucial role of acquired cultural values when choosing a pot in the market, and how some readily identifiable features such as colour and finish are taken as signifiers of the 'reputable pot' – the pot which you know that 'works' –, and ultimately determine the choice. For most Andean peoples, for example, the red colour is taken as an indication that the pot is strong, 'well fired', and this leads some potters take extra care in adding a red slip or salt glaze to make sure that their pottery is red. For 16th-century Oberstockstall, the extent to which these associations were conscious or unconscious, as well as the precise set of qualities related to the immediate appearance of the crucibles, remain to be clarified in more detail. Nonetheless, this seems a feasible explanatory framework, which takes into account the interactions people-people and people-artefacts (cf. Schiffer and Skibo 1997: 31). Furthermore, Sillar's study pointed out the distinctive standardisation and conservativeness of the various pottery producing communities, each marketing a limited range of 'trade-mark' designs – even when capable of making others – that hold their respective reputation. A result of this is that the customer, even when often unaware of its provenance, identifies a pot as a part of a group of wares previously used

and possibly liked. Again a parallel with the Oberstockstall case appears tempting since, as will be discussed in chapter 10, the 'triangular, black, smooth' crucibles quite probably had to compete in the market with another major group of crucibles, also triangular, but rough in surface and bright in colour.

The formal properties of an artefact are a product of technological choices, and it makes little sense trying to separate those out as either function or style, since these are not mutually exclusive categories (Schiffer and Skibo 1997; Sillar and Tite 2000). Behind different choices there is a range of reasons, conditioned by material factors as well as by contingent social, cultural and historical dynamics; reasons related to the artefacts themselves but also to those producing, selling, buying and using them. What a producer intends or what an end user expects, are very much dependent on factors outside what we may consider purely technical needs – even these needs are relative to specific contexts, and are very likely to change through time. In another study supporting this view, David Gaimster and Beverley Nenk (1997) have persuasively argued that an important driving force behind the transformation of the English ceramic market between 1450 and 1550 was a process of social emulation. As ceramic utensils, fixtures and fittings acquired a central role in the households, lower middle classes aspired to emulate a cosmopolitan elite that displayed their 'Continental Renaissance culture', using pottery as a principal medium. This prompted a rise in imported wares, but also an increased diversity in the native ceramic production. Focussing on post-medieval ceramics from Yorkshire and Humberside, Chris Cumberpatch (1997b; 2003) has attempted to diversify this picture by stressing that not only the emulation of continental goods but also intrinsic cultural factors and customers' expectations played a part in the transformation of the English ceramic tradition, although this study failed to identify specific alternative motivations. In any case, these approaches evince that appearance matters, and that this is a product of a "recursive" and "reflexive" negotiation which depends on the social context while exerting an influence on it (Cumberpatch 1997a; 1997b). If this was the case for domestic pottery, then, surely, symbolic and cultural issues must have been at stake in the production and consumption of the pottery used in such notional a world as chymistry.

6.2.4. Crucibles at use: more on performance

The above discussion of the vessels' appearance and the way they would be perceived by potential consumers relates to the wider performance of the crucibles: how the crucibles are perceived and, eventually, whether they are purchased, is conditioned by technological choices but ultimately depends on the specifics of the social, cultural and economic context. Similarly, the performance of the crucibles while at use at the laboratory ought to be addressed.

Some of these aspects have already been discussed. For example, it was mentioned above that the flat base of the crucibles can be related to their use in a furnace with indirect firing, as is typical for fire assays. However, it should be noted that the crucibles did not normally sit directly on the furnace plate, but on bricks or "crucible bases" such as those mentioned by Ercker (e.g. Sisco and Smith 1951: 180) and illustrated by Biringuccio (e.g. Smith and Gnudi 1990: 292-293, figs. 45 and 46). In Oberstockstall, many of the crucibles show evidence of use while sitting on a thick lump of clay or a brick of little refractoriness, which turned into a viscous paste under the high temperatures (fig. 55). It is likely that this flexible base would enhance the vessels' stability, as some operations required stirring, or the addition of new material to the charge, whilst the crucible remained in the furnace. If a vessel had to be temporarily taken out of the furnace, for example to remove a sample and check the evolution of the process (e.g. Sisco and Smith 1949: 141), the viscous base would alleviate the possible impact of the hot crucible on the hard furnace plate when the vessel was put back into the chamber. Finally, this base would also facilitate a more even heat transfer, while lifting the crucible to the upper and hotter area of the furnace chamber. Overall, this peculiarity of the vessels' use would minimise the potential risk of

Figure 55. Example of a vessel sitting on a less refractory 'crucible base'.

these flat-bottomed crucibles failing at what could be one of their weak points: the angular inflection between base and body (cf. Tite *et al.* 2001; Tite and Kilikoglou 2002).

Ercker clearly states that he prefers three-legged vessels "because the heat can easily reach the thick bottom without there being any obstacle, so that the silver or other charged metals heats up twice as fast as in those crucibles that have to be put on a thick base" (Sisco and Smith 1951: 180). While this may be true, these would require a more intricate manufacture, probably raising the price of the final product in the market, and perhaps creating structural weaknesses at the joints of the three feet. For the assay crucibles from Oberstockstall, the use of a thick viscous base seems to have been an easy and useful technical solution: only in one of the crucibles examined has the base catastrophically come off – significantly, after pouring some substance, which again shows the stresses to which these vessels were subjected when manipulated while hot.

Microscopic and SEM-EDS analyses of used crucibles provide further information about their performance during use. For example, crucibles OB 345, OB 494 and OB 515 seem to have been used for the primary fluxing and reduction of fahlore, a complex sulphidic ore for which the generic formula is $(Cu,Ag)_{10}(Fe,Zn)_2(As,Sb)_4S_{13}$, traditionally used as a copper and/or silver ore. In all cases, the slag layer has a strong component of molten ceramic material, but is enriched in lime, potash, phosphorus, iron, soda, magnesia and chlorine. It contains some silica, potassium feldspar and apatite grains, as well as metal droplets of lead and antimony alloyed in varying proportions, sometimes bearing minor amounts of copper, arsenic, tin, sulphur and iron. Towards the bottom of the crucible, the presence of metallic elements increases, especially that of lead. This suggests that this metal was added to the charge to function as a collector for the silver – perhaps as an oxide, which would flux the charge and, once reduced, would collect the noble metals. The positive correlation between sodium and chlorine insinuates that rocksalt (NaCl) could have been added as a further flux, together with sand (fig. 56).

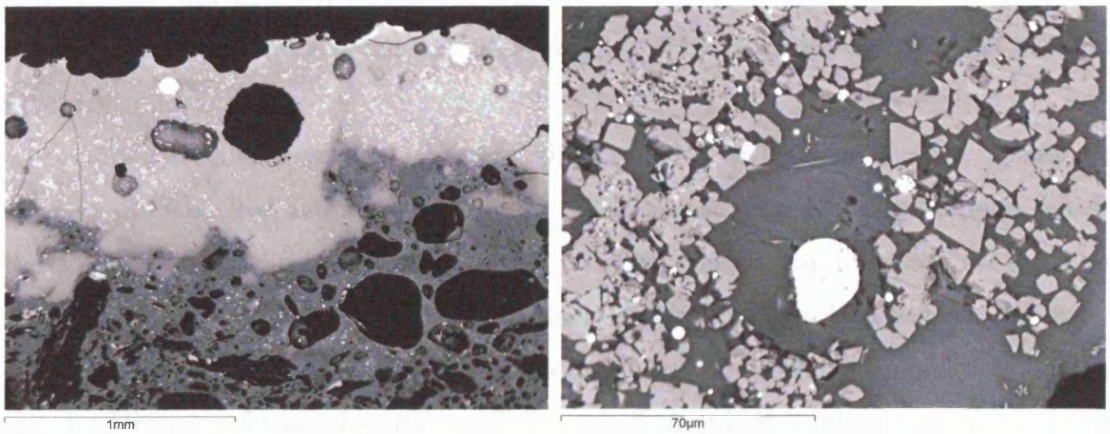


Figure 56. Two BSE micrographs of a crucible that was probably used for assaying fahlore. Left, interface between the highly vitrified graphitic fabric (bottom) and the slag layer. The slag consists mostly of iron oxide and silica (each >30 wt%), with alumina (11 wt%), lime (8 wt%), soda (4 wt%), potash (4 wt%) and chlorine (<1 wt%). One of the bigger metallic prills (bright globules) is antimony (84 wt%) with copper, lead and arsenic in smaller amounts; the other one is copper (78 wt%) with antimony and sulphur. Right, detail of the slag layer, showing numerous crystals of iron oxide (light grey) and metal prills (bright). The biggest prill is copper sulphide, containing iron and antimony. The rest are varying alloys of antimony and copper, containing traces of iron and sulphur (OB 494/s1, left at 50x, right at 800x). See Appendix 2 for detailed analytical results.

In any case, what really matters for the purpose of this study is not the specific utilisation of the crucible but rather the conditions of use. In the above cases, it is apparent that the vessels were subjected to very high temperatures over extended periods. Their fabrics show continuous vitrification, with development of fine to medium voids (fig. 57). Most feldspar and iron silicates within the fabric appear to have become molten, fluxing the ceramic surrounding them and hence leaving some softer, more vitrified areas within the crucible wall (fig. 58). In some samples, this phenomenon occurs with the minerals attached to graphite as well. Even the more refractory quartz grains appear sometimes decomposing.

These operations were carried out in strongly reducing conditions, probably with the crucibles covered. This was a necessary requirement of the operation, aimed at reducing the metal components of the mineral, possibly prior to extracting the noble metals by further concentration, scorification and/or cupellation. The reducing atmosphere within the crucibles is shown by the fact that most of the graphite has remained unaltered – only the smallest inclusions have disappeared, particularly towards the outer surfaces – but also, importantly, by the presence of many tiny globules of iron sulphide scattered within the ceramic body. As the only escape for the sulphur vapour was through the ceramic fabric, it reacted with the iron oxide of the ceramic, which precipitated as iron sulphide (figs. 57-59). The close contact with carbon from the

graphite would facilitate this reaction. Accordingly, the concentration of free iron oxide in the ceramic matrix was decreased (see Table 1, p. 113, crucibles OB 345, OB 494 and OB 515).

Several considerations regarding the crucibles' performance arise. Firstly, it appears obvious that fabric refractoriness was a major technical requirement, which these vessels served satisfactorily. Whilst, at first, one might interpret the melting minerals within the fabric as a flaw, potentially making the crucibles weaker and more prone to failing, it should be noted that none of the hundreds of crucibles examined appeared catastrophically bloated or thermally distorted. Possibly, in relatively small sizes (the diameter of the 'fluxed' areas is never larger than 0.5 mm) and numbers, as these minerals dissolved into the ceramic matrix, they increased the viscosity of the paste and helped retain its rigidity during use (cf. Freestone 1989: 158, where this phenomenon is suggested for quartz temper; and Freestone 1991, for an example of crucibles where this local 'fluxing' was created by adding crushed glass to the fabric). More refractory inclusions such as quartz and graphite would, at the same time, help keep the crucibles' strength.

Secondly, the role of graphite as a reducing agent becomes apparent. A reducing atmosphere was a requirement for the graphite not to burn out. However, at the same time, graphite itself would contribute to keep conditions reducing within the vessel. In this process, as the iron oxide within the fabric reacted with the sulphur-rich vapour and the carbon, it precipitated as discrete particles of iron sulphide. The presence of iron oxide as a potentially fluxing agent in the ceramic was thus minimised, which further enhanced the thermal refractoriness of the vessel. In another crucible analysed (OB 466), part of the iron oxide within the fabric was reduced to the metallic state during use, a phenomenon also documented in protohistoric pottery from Italy (Bertelle *et al.* 2000) and 19th-century steel making crucibles from India (Freestone and Tite 1986).

A completely different case was that of crucible OB 495. Here, the reaction was oxidising and most of the graphite burned away – only part of it remained in the core of the wall. The most abundant contamination on the inner surface is by iron oxide (~28 wt%), together with alumina and smaller concentrations of oxides of lead, sulphur and arsenic. At temperatures similar to those recorded in the crucibles discussed above, this vessel would have been more prone to catastrophic bloating. However, the reaction took place at lower temperatures and possibly for a shorter period, as shown by in the limited degree of vitrification in the matrix (fig. 60) and, more particularly, in the lute

adhering to the outer surface (fig. 107, p. 181) together with the fact that most silicate inclusions remain unaltered. As a consequence, the performance of the vessel was acceptable. Crucible OB 520 shows similar conditions of use, with relatively low vitrification and a substantial loss of graphite (fig. 61).

These instances demonstrate the need to observe the specific conditions of use of a given crucible and its particular response to these – *i.e.* its performance –, in order to draw archaeologically significant conclusions. In particular, one may revise the usual consideration of the iron oxide content as a detrimental factor when comparing the thermal refractoriness of different ceramics. We should be wary of the limited applicability of general comparisons of ceramics' material properties, detached from their contexts[18].

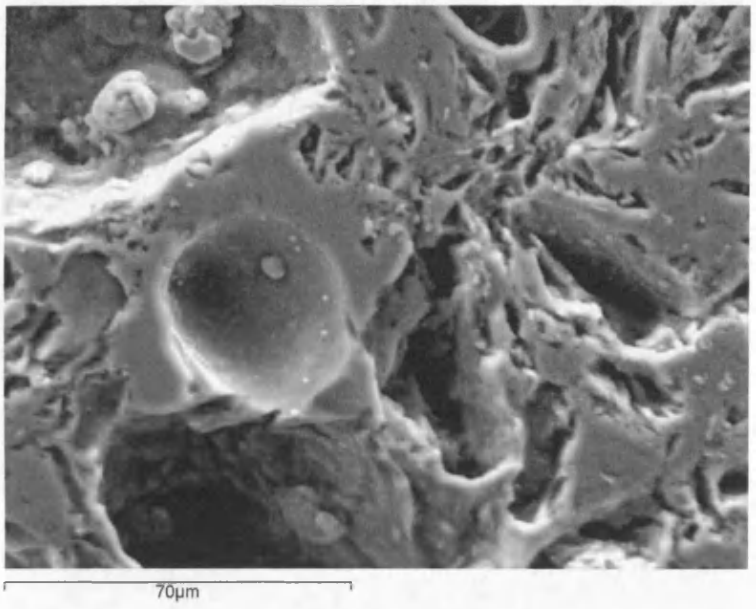


Figure 57. SE photomicrograph of a used crucible, showing complete vitrification and development of voids, some of them in the spaces previously occupied by very small graphite speckles. The light globules are of iron sulphide (OB 345/s1, 800x). Compare to fig. 40 (p. 114).

[18] While the use of the crucibles in a reducing atmosphere is seen here as a factor prompting the reduction of iron oxide to metal and thus increasing the heat resistance of the vessel, experimental studies (Tite *et al.* 1982b) have indicated that reducing conditions may favour the reduction of ferric iron in the clay to the more reactive ferrous form, thereby initially decreasing the refractoriness of the ceramic. This again stresses the need to investigate the vessels' performance in specific circumstances, rather than material properties alone.

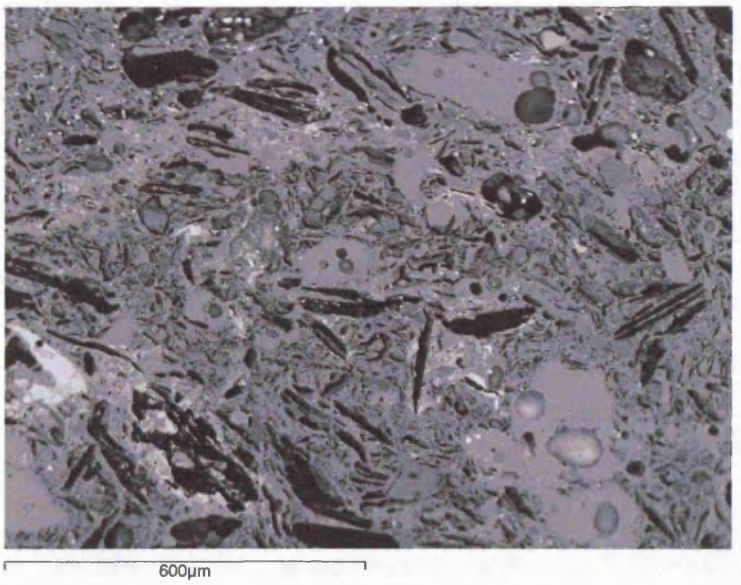


Figure 58. BSE photomicrograph of a crucible that was used under reducing conditions. The more vitrified (glassy and with round voids) areas are the result of less refractory silicates melting and fluxing the surrounding matrix. All the brightest phases are globules of iron sulphide (OB 345/s1, 50x). Compare to fig. 41 (p. 114).

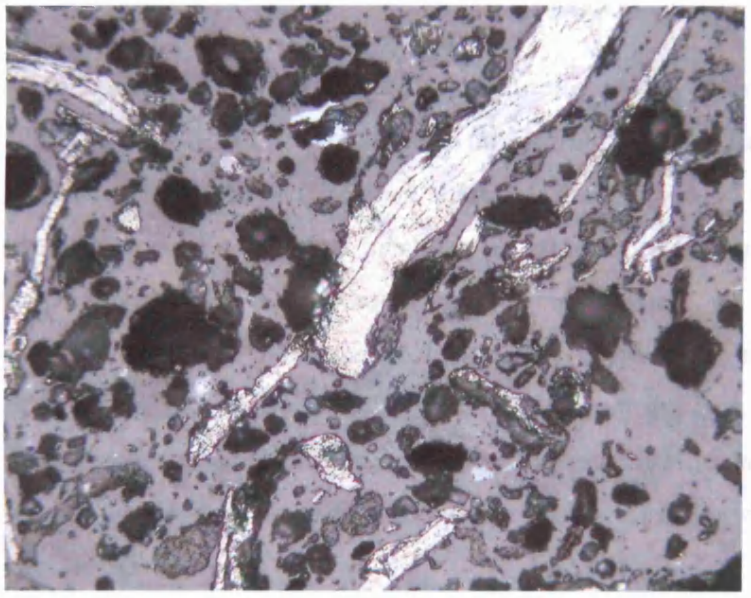


Figure 59. Detail of the fabric of a crucible used under reducing conditions. Note the complete vitrification of the fabric, with closed porosity, but the considerable amount of remaining graphite inclusions (OB 494/s1, PL, 200x).

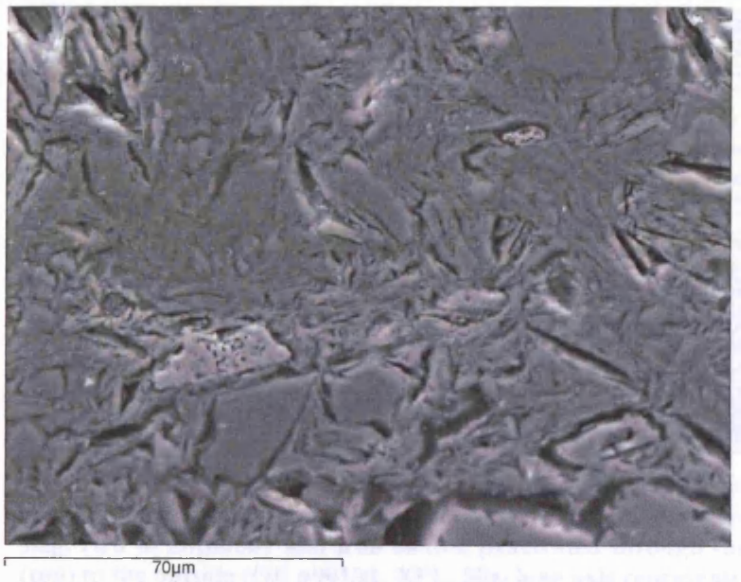


Figure 60. SE photomicrograph of a crucible that was used under oxidising conditions, showing the voids left by burnt-out graphite inclusions, and an extent of initial vitrification similar to those of unused crucibles (OB 495/s1, 800x). Compare to figs. 40 (p. 114) and 57 (p. 136).

Figure 61. Detail of the fabric of a crucible used under oxidising conditions. Note that, in spite of the relatively low vitrification, graphite inclusions have burned out, leaving elongate voids within the matrix. Compare to fig. 59 (OB 520/s1, XPL, 200x).

Another factor at issue during the use of the Oberstockstall crucibles seems to have been the resistance to chemical attack. As mentioned above, several crucibles show evidence of corrosion by the hot charge. It was also noted that the surface smoothness, together with the chemical inertness of graphite inclusions, may have helped hamper this. However, we do document some samples where the penetration of metal oxides into the fabric led the vessel to failure. This is the case, for example, of crucible OB n001, where a thick layer of slag, rich in antimony and iron oxides (~20 wt% each), and with smaller concentrations of oxides of magnesium, sulphur, potassium, calcium, copper and lead, penetrated through the wall and exuded through a crack on the outer surface (fig. 62).

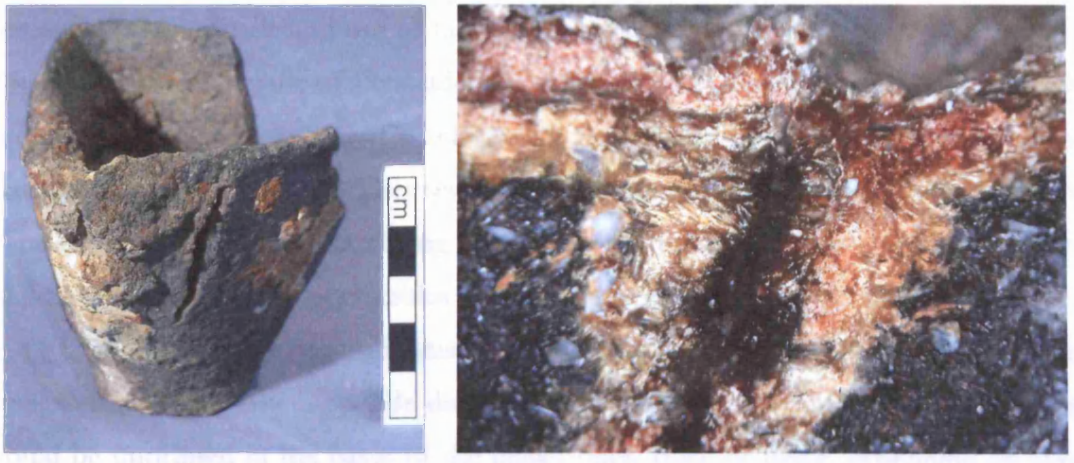


Figure 62. Examples of crucibles affected by chemical attack. Left, crucible where one pouring spout has been corroded (OB 315). Right, photomicrograph of a crucible where the slag, rich in antimony and iron oxides, penetrated through the body from the inner surface (top) to the outside (OB n001/s1, XPL, 50x, long axis represents ~2 mm).

Hence, while thermal refractoriness seems to have been a technical requirement with which the crucibles in Oberstockstall dealt successfully, it seems that a sufficient chemical refractoriness was more difficult to achieve. A major concern of 16th-century assayers was obtaining strong fluxes, often expensive and difficult to produce, as evidenced in the attention paid to this topic in the foremost treatises (e.g. Hoover and Hoover 1950; Sisco and Smith 1951). Just as some fluxes and reagents were very effective in facilitating the decomposition of the crucible charge, they could also lead to the degradation of the fabric of the vessel itself.

Finally, an important aspect directly affecting the performance of the crucibles would have been the number of use cycles, as attributes such as the chemical, thermal or mechanical stability would be increasingly challenged with repeated uses. Analyses so far do not clearly indicate that any of these crucibles was used more than once, although this remains feasible. In the assemblage from Weyerstraße in Cologne introduced earlier (section 4.10, see also 9.3), Rehren (1996a; 1996b) identified a triangular crucible with two distinct slag layers denoting two different stages of utilisation. If fire assays aiming at quantitative inferences were conducted, one would expect new crucibles to be used in order to avert cross-contamination. Nevertheless, for other operations not requiring so much accuracy, crucibles were probably re-used. It is hoped that further work on the residues within the Oberstockstall vessels will shed more light on this question.

6.2.5. The manufacture and performance of the crucibles: a summary

The information presented in this chapter allows a general reconstruction of the process of manufacture and use of these technical ceramics. The crucibles recovered in Oberstockstall were made of a considerably refractory clay, rich in alumina and normally containing graphite inclusions. This clay was levigated, and finer clays were reserved for progressively smaller vessels. They were modelled on the potter's wheel in the shape of a truncated cone, the rim then being folded into the triangular shape. This would have facilitated the production of crucibles in large numbers.

Once the modelling had finished, the crucibles would be left to dry for some time, before turning them up side down. At this point, a ceramic die with a 'T' stamp would be imprinted in the bases of the bigger ones, once or twice, depending on their size or value. Possibly, it was also now when the crucibles were wiped to make their surfaces particularly smooth.

Once the drying was complete, the vessels were fired in a moderately reducing atmosphere, at temperatures between 950 and 1050 °C. Towards the end of the firing, the kiln would be choked with organic matter in order to create a smoky atmosphere and thus furnish the crucibles with a black lustre. It is suggested that this concern with the appearance of the crucibles may respond to a widespread conception of matter in the Renaissance, whereby the properties of matter are affected by its outward quality, rather than by its composition. Alternatively, the specific appearance may constitute a 'marketing strategy' or trade mark.

The resulting crucibles, particularly those containing graphite, were thermally and chemically refractory, resistant to thermal shock and they showed good tensile strength. However, their performance was ultimately affected by their specific conditions of use, as well as the number of uses. For example, it appears that the relatively high iron oxide content in the ceramic matrix would be detrimental for long reactions in oxidising conditions, but not necessarily so if the atmosphere was reducing and/or the operation short. The crucibles were often used sealed or lidded, and sitting on a crucible base, as typical for fire assays.

6.3. The scorifiers

The scorifier is another versatile tool frequently found in archaeological laboratories and workshops. However, in contrast to the wide variety of contexts of use documented for triangular crucibles (see chapters 7-10), the utilisation of scorifiers seems to be constrained to fire assay and refining activities, even thought the final intent of these might differ. Formally, the scorifier is simply a ceramic dish (fig. 63). As the name indicates, these were predominantly used for scorification processes, *i.e.* to transform the unwanted components of a sample into slag, in a typically oxidising reaction. These operations could be, for example, the roasting of a mineral, the primary melting of a metal or ore sample with some flux, or the oxidation of lead bullion in order to pre-concentrate the noble metals prior to cupellation. The combination of raw materials and additives that could be melted together in a scorifier for assaying purposes is practically endless (cf. Unglik 2000), which makes the analytical identification of specific recipes very difficult.

Figure 63. Group of scorifiers from Oberstockstall. Scalebar is 15 cm.

The first archaeologically documented scorifiers were presented and analysed by Richard Pittioni (1975; 1978; 1985). Originally, it was not uncommon to term them "heating trays" (Roesdahl 1977, as cited in Bayley 1992: 749; Bayley and Barclay 1990; Söderberg 2004), mostly owing to doubts about their interpretation. Later, once it was proved that they could be used for the refining of noble metals by oxidation of a lead bullion, some researchers referred to them as "cupels" (Bayley 1988; Éluère *et al.* 1989; Bayley 1992; Sperl 1996; Bayley and Eckstein 1997). Recently, the more neutral designation "saucer-shaped ceramic dish" has been used (Bayley 2003). While the term "cupel" may be acceptable for medieval and earlier periods – before the use of ash-

based cupels was established –, "scorifier" is preferred as a wider term that denotes dishes that could be used for cupellation as well as for other scorification processes[19]. Since the 16th century, scorifiers and cupels appear as clearly different pieces of equipment in the written sources, hence this separation is kept for the present study[20].

Medieval and later scorifiers have been found in a variety of metallurgical workshops across Europe, including Scandinavia (Söderberg 2004 and references therein), England (Tite *et al.* 1985; Bayley 1988; 1992; 2003; Freestone 1989; Bayley and Barclay 1992), Germany (Eckstein *et al.* 1994; Rehren 1996a; 2002a; Shifer 1998), Switzerland (Kamber *et al.* 1998) and Austria (Pittioni 1975; 1978; 1985; Sperl 1996). Regarding the sizes, Agricola recommended a diameter of three digits [~55 mm] (Hoover and Hoover 1950: 228), but archaeological examples range from 30 to 200 mm.

6.3.1. Formal properties

Most of the approximately fifty scorifiers from Oberstockstall fall within two formal types, one deeper – with a diameter to height ratio in the range 2-3 – and one shallower – with a ratio between 4 and 6. The first group consists of dishes of a very standard shape, with very thick bottoms and diameters of either ~55 or ~70 mm at the rim. The second group has thinner bodies, and their diameters range between ~65 and ~200 mm (von Osten 1998) (fig. 64). All of them appear to have been wheel-thrown, as suggested by the curved rotation marks seen in some bases. Finally, there are at least three broken sherds that were shaped into a subcircular shallow dish, similar to the scorifiers, although the traces of use in these are not as substantial as in most scorifiers[21].

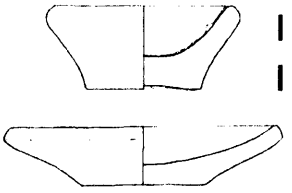


Figure 64. Two main formal types of scorifiers documented in Oberstockstall (drawings from von Osten 1998). Scalebar is 3 cm.

[19] Bayley and Eckstein (1997) suggest that, before the 16th century, scorifiers could be used in successive oxidation stages culminating in the final refining, thus there would not be a clear cut distinction between scorification and cupellation. While this is very likely, I suggest that the term scorifier be maintained, to differentiate this from the more specialised bone ash cupel, which was only used for cupellation.

[20] Ercker differentiates between crucibles, scorifiers and (ash) cupels or tests as distinct tools. In Agricola's work, instead, triangular crucibles, scorifiers and cupels all appear as subtypes of the generic category 'crucible'. In any case, the ceramic plate is clearly identified as a specific instrument with various applications, rather than as a 'ceramic cupel'.

[21] The recycling of broken sherds for scorification purposes has also been documented in medieval contexts (e.g. Bayley 1992).

Unfortunately, none of the scorifiers in the assemblage seems unused: even those with less conspicuous traces of use showed increased concentrations of iron and lead oxides when their inner surfaces were analysed by ED-XRF. For this reason, it is difficult to estimate the original appearance and vitrification of the vessels. In their current state, their fabrics normally show a buff to orange colour, although there are some samples with grey surfaces, particularly those with lighter traces of use. The slag attached to them is usually a relatively thick (1-8 mm), bright, black layer, often with a ~30 mm wide depression in the centre. In addition, there are scorifiers where this slag is green or yellow and with variable amounts of big inclusions (up to 2 mm), and others with just some evidence of burning on the inner surface (fig. 65).

Figure 65. Inner surfaces of different scorifiers from Oberstockstall, showing variable traces of use. Not to scale.

Microscopic and chemical analyses allowed the identification of three different fabric types. It should be noted from the outset that no correlation was found between fabric and formal types, or between these and clearly distinct traces of use.

The first fabric type is very similar to that of the graphitic triangular crucibles (see above, section 6.2). It consists of a fine-grained matrix containing about 30 vol% of graphite (sometimes intergrown with silicates) and 15 vol% of silicate inclusions. These

are subangular and moderately spheroid, not very well sorted but normally below 0.3 mm in diameter. Contrary to the crucibles, where quartz and potassium feldspar appear in similar proportions, feldspar grains within these scorifiers' matrices are more abundant than quartz. The paste also contains some amphibole, discrete iron oxide, rare plagioclase, apatite and mica, as well as very small ilmenite, rutile and zircon inclusions (figs. 66-67). The chemical compositions of these matrices fall within the same range as the triangular crucibles. The highest variations appear in soda and potash concentrations, but these may be related to varying concentrations of silicate inclusions and contamination from the charge (Table 4, fabric A).

The second major ceramic type is characterised by the abundance of quartz inclusions, of about 40 vol%. These inclusions, normally below 0.3 mm large, are subangular, with a moderate degree of sphericity, and not very well sorted. Some feldspar grains are also present, together with iron oxide, amphibole, and tiny ilmenite, rutile and zircon grains. Besides, this fabric type contains some inclusions (≤10 vol%) that may be interpreted as grog (figs. 68-69). These inclusions, normally oblong and with a maximum diameter of 0.5 mm, usually appear denser and more vitrified than the main ceramic matrix, and are surrounded by a shrinkage void; they tend to show internal alignment, although internal fractures are not always present; they often contain small silicate inclusions and, compositionally, they are either very similar to the main body or richer in iron oxide (fig. 70). While none of these criteria can be conclusive for the identification of artificially added grog as different from natural argillaceous inclusions (cf. Whitbread 1986; Wheeler 2003), it seems reasonable to accept them as grog. Further arguments to support this interpretation are, on the one hand, the strong resemblance of this ceramic paste to that of the graphitic scorifiers – which, however, do not contain this kind of inclusion – and, on the other hand, the historical (Norton 1652: 92; Sisco and Smith 1949: 111; Hoover and Hoover 1950: 230; Hawthorne and Smith 1979: 142) and archaeological (Freestone and Tite 1986; Martinón-Torres 2001) evidence for the use of grog in metallurgical ceramics. The chemical composition of the main ceramic matrix in these plates is very similar to that of the previous type, albeit with lower titania concentrations (Table 4, fabric B).

Finally, the third ceramic type is represented by a single big scorifier (OB 269, 130 mm diameter), containing abundant, poorly sorted quartz (~30 vol%, ≤1 mm) and grog, together with some potassium feldspar, amphibole and calcium minerals (figs. 71-72). The composition of the ceramic matrix, when compared to the others, stands out

primarily in the lower alumina and the higher lime and magnesia concentrations, the latter mostly due to minerals clearly decomposing within the paste (Table 4, fabric C).

	Fab.	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO
OB n006	A	1.0	0.3	32.2	54.3	0.2	0.5	0.1	5.9	0.5	1.1	3.9
OB 279	A	0.3	0.7	30.5	56.8	0.3	-	-	2.0	0.8	1.5	7.1
OB 284	A	1.0	0.4	33.4	55.7	0.3	-	-	2.0	0.5	1.3	5.4
OB 268	B	0.2	0.7	33.2	58.9	0.3	-	-	1.8	0.6	0.6	3.6
OB 276	B	0.3	0.4	31.4	59.6	0.1	-	0.2	1.9	0.7	0.7	4.8
OB 277	B	0.2	0.5	32.3	59.2	0.2	-	-	1.6	0.7	0.7	4.5
OB 281	B	0.2	0.4	32.0	59.7	0.3	-	-	1.9	0.6	0.5	4.6
OB 282	B	0.3	0.5	30.1	61.6	0.3	-	-	2.1	0.6	0.6	4.1
OB 269	C	0.4	1.0	26.1	63.1	0.3	-	-	1.8	2.2	0.8	4.3
Crucibles		0.2	0.7	32.4	57.3	0.3	-	-	2.1	0.7	1.3	5.0

Table 4. Average composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of the different ceramic fabrics identified in the scorifiers from Oberstockstall. The average composition of the crucibles is also shown for comparison. See Appendix 1 for full data.

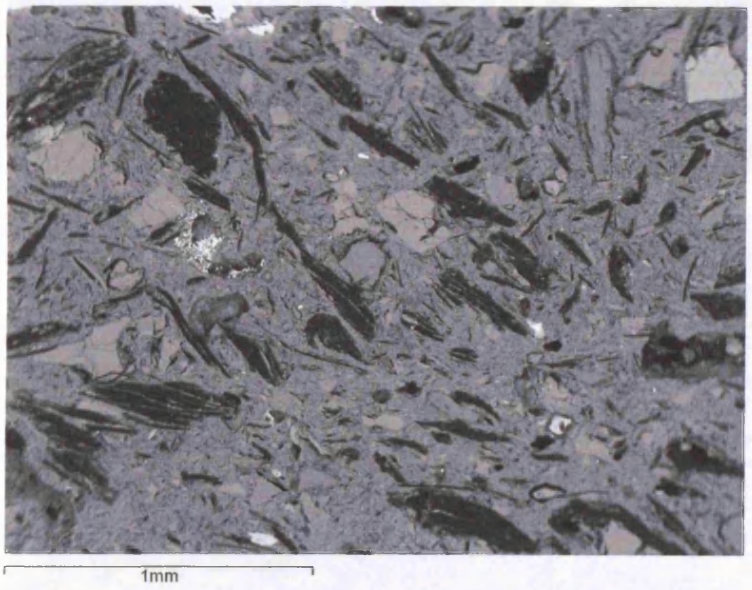


Figure 66. BSE photomicrograph of a graphitic scorifier (fabric A) at low magnification, showing abundant graphite inclusions as well as silicate minerals including abundant feldspar (light grey) (OB n006/s1, 50x). Compare to fig. 41 (p. 114).

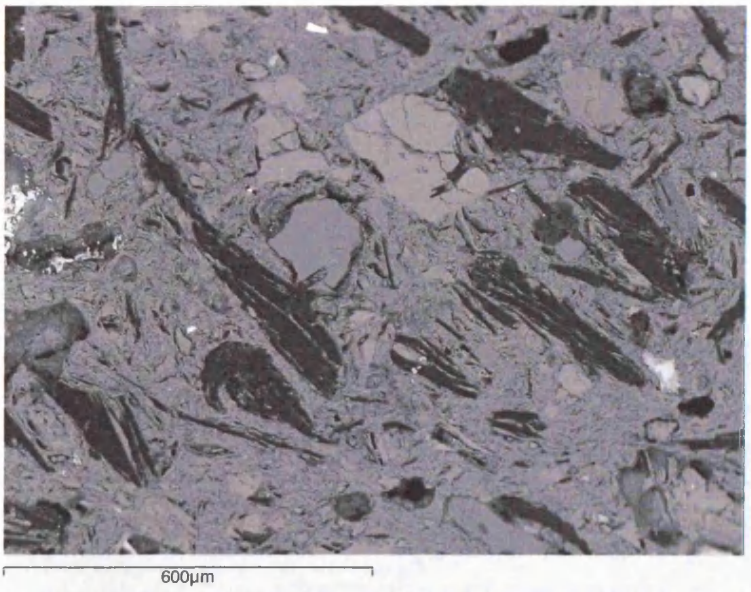


Figure 67. BSE photomicrograph of a graphitic scorifier (fabric A) at higher magnification (OB n006/s1, 100x). Compare to fig. 47 (p. 116).

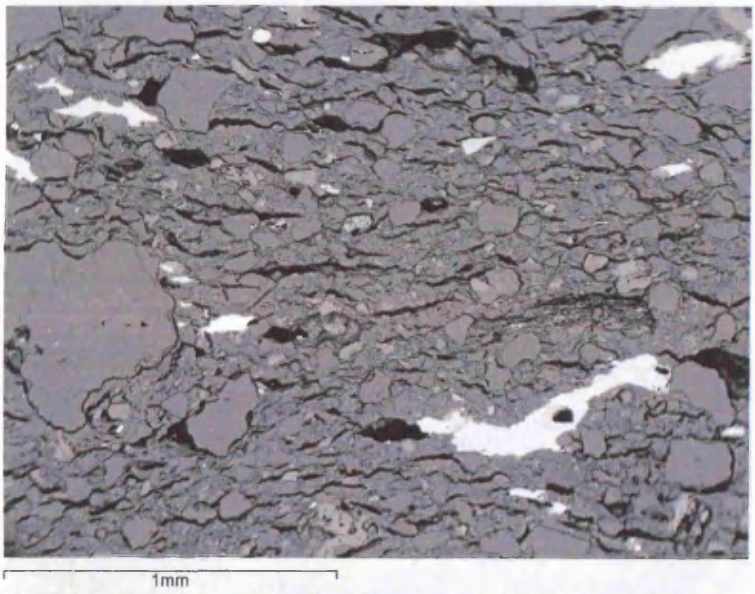


Figure 68. BSE photomicrograph of a non-graphitic scorifier (fabric B) at low magnification, showing abundant quartz inclusions and elongate cracks. The white phases are slag penetrating into the ceramic body (OB 268/s1, 50x).

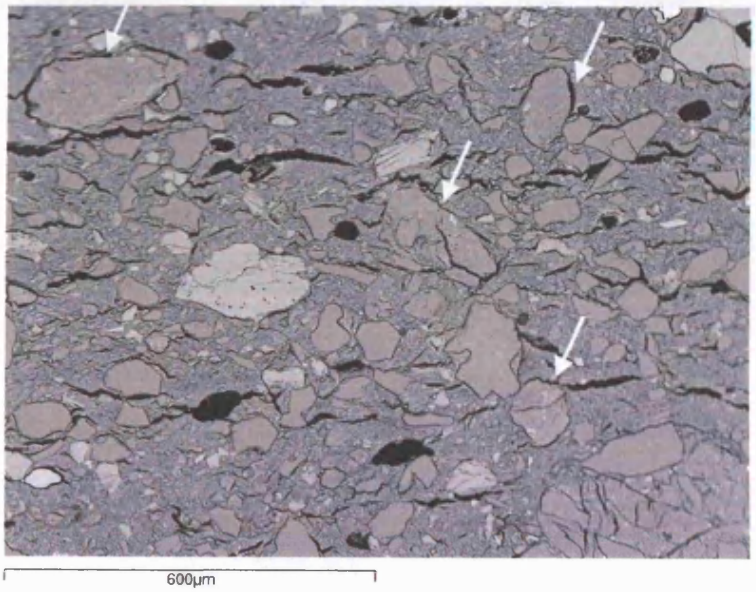


Figure 69. BSE photomicrograph of another non-graphitic scorifier (fabric B) at higher magnification, where some possible grog temper (arrows) can be noticed, together with the silicate inclusions (OB 276/s1, 100x).

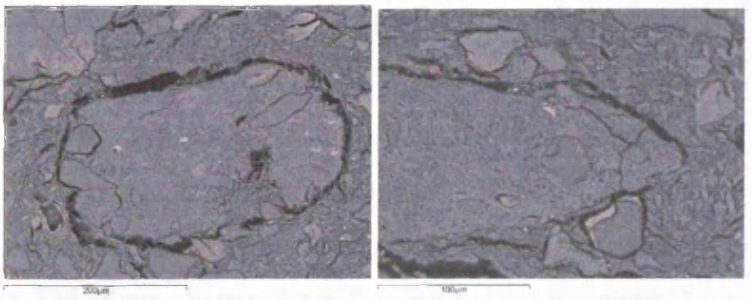


Figure 70. Detail of some possible fragments of grog within the fabric of a scorifier (OB 276, BSE, left at 300x, right at 500x).

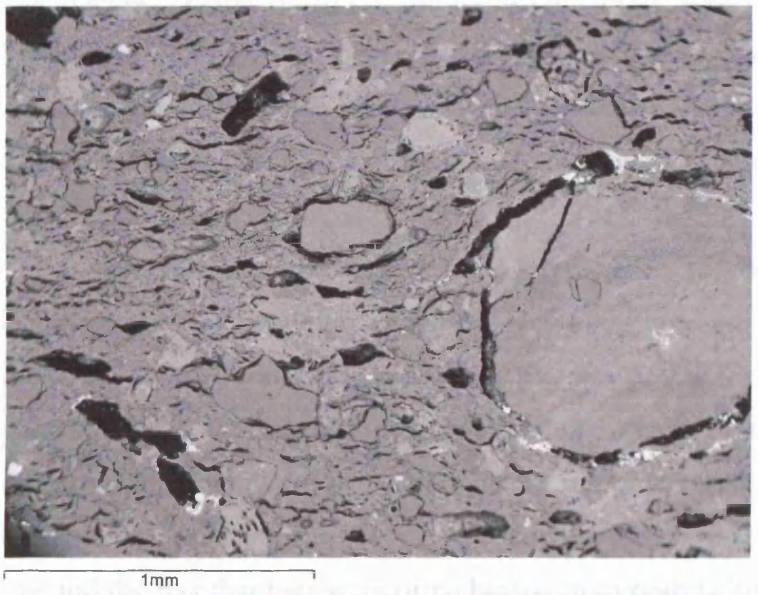


Figure 71. BSE photomicrograph of the ceramic fabric of the scorifier OB 269, a distinct type within the assemblage (fabric C), where a big grog inclusion can be noticed (right), as well as smaller silicate inclusions surrounded by shrinkage voids and elongate cracks (OB 269/s1, 50x).

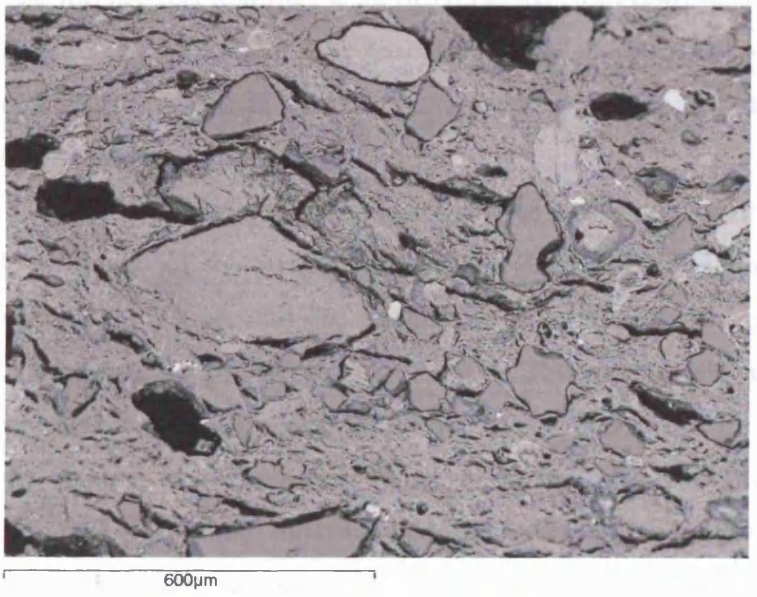


Figure 72. Detail of the fabric of OB 269, with grog temper and silicate inclusions (primarily quartz – dark grey). Shrinkage voids can be noticed around the inclusions, as well as elongate cracks across the body (OB 269/s1, 100x).

6.3.2. Material properties and performance characteristics

Before addressing the material properties of the ceramic pastes, and how these relate to performance, the most characteristic formal feature of the scorifiers should be noted, *i.e.* the shallow, open shape. This makes them clearly different from the crucibles, in that the former would normally favour an oxidising operation, whereas the latter would allow a better control of the redox conditions. Nevertheless, even if no evidence of this practice has been observed in Oberstockstall, it should be noted that Ercker documents the use of two scorifiers – one upturned and luted onto the other as a lid – for the cementation of gold in necessarily reducing conditions (Sisco and Smith 1951: 184-186).

The material properties of the graphitic fabric are obviously the same as discussed for the graphitic crucibles. However, they are not equally relevant. The improved thermal conductivity, for example, was probably not a crucial factor affecting performance. Whilst, theoretically, graphite might enhance thermal diffusion across the fabric and thus minimise thermal gradients across the wall, it is believed that it would not make a significant difference in the case of the scorifiers, given their generally small size and the fact that they were often heated from both below – from the fire box – and above – by bellowing into charcoal within the chamber – simultaneously. Similarly, considering the small weight of the samples processed, tensile strength may not have been a major requirement.

The likelihood of graphite acting as a reducing agent during scorification is very low. In one of the analysed specimens (OB 279), the internal surface of the ceramic appears dark grey, and the graphite inclusions are better preserved, thus indicating a locally reducing atmosphere (fig. 73, left). This may be related to the presence of a dense and thick slag layer within the vessel, which prevented the penetration of oxygen into the ceramic from the inside. However, in the other two graphitic specimens examined (OB 284, OB n006), most of the graphite has burned away, leaving big elongate voids within the paste that were further developed during vitrification (fig. 73, right). Since scorification typically is an oxidising process, the burning away of graphite was presumably a usual phenomenon. Ercker states on several occasions that the reactions carried out in scorifiers were meant to be relatively quick, usually taking half an hour and never more than one hour (e.g. Sisco and Smith 1951: 38-39). Seemingly, this interval was long enough for the graphite to disappear, in a process that would render a

weaker scorifier – no longer benefiting from the material advantages of graphite inclusions, and conversely suffering from big porosities that would favour the penetration of the slag into the ceramic (cf. Freestone 1989: 158). It follows that the presence of graphite in the fabric might have been advantageous only during the early stages of the process, and depending on the specific conditions of use.

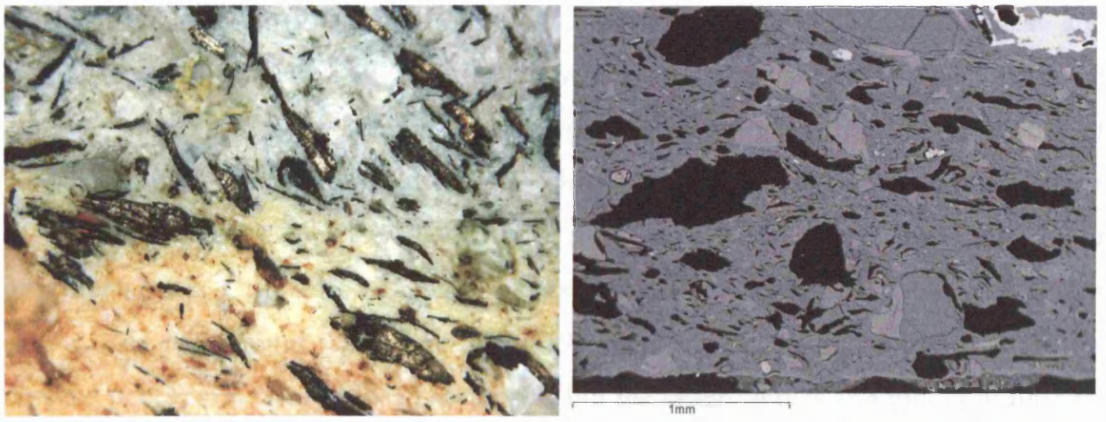


Figure 73. Left, fabric of a graphitic scorifier, where the grey colour and larger amount of graphite remaining denotes locally reducing conditions towards the slagged inner surface (top, not in the image) (OB n006/s1, XPL, 50x, long axis represents ~2 mm). Right, fabric of another graphitic scorifier, where most of the graphite has burned away in an oxidising atmosphere, leaving large voids (OB 279/s1, BSE, 50x).

Other properties may have been more important, particularly thermal shock resistance and toughness. The slag layers within all three graphitic scorifiers analysed show abundant round voids that 'froze' as the plate was apparently removed from the furnace while hot (fig. 74). This is in contrast to the rest of the scorifiers, where the slag is much denser and often shows big crystals developed whilst slowly cooling down, probably inside the furnace (fig. 75). Both possibilities are accounted for in the written sources, depending on the specific reactions conducted (cf. Sisco and Smith 1951: 38-39). It would thus appear tempting to think that graphitic scorifiers were reserved for those operations involving higher thermal stresses. Nonetheless, all of the plates may be assumed to have been subjected to thermal and mechanical stresses at some stage, since they were normally

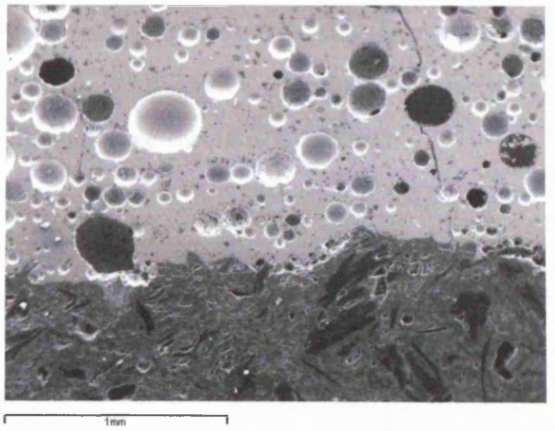


Figure 74. Example of a graphitic scorifier that was removed from the furnace while hot, hence the round voids 'frozen' within the slag (OB n006/s1, BSE, 50x).

placed in the furnace only when this was hot, they often had to be temporarily removed and put back in, new components were added to the charge during the process, and stirring was a usual requirement.

Those fabrics not containing graphite, but having instead abundant quartz and some grog, would also perform efficiently in the face of thermal contrasts and mechanical impacts. All the specimens analysed clearly show the elongate cracks and shrinkage voids characteristic of quartz-tempered fabrics (see above, figs. 68-72), which would arrest and dissipate otherwise catastrophic fractures (cf. p. 120). The presence of grog might also enhance the resistance to chemical attack more efficiently than quartz grains (Freestone and Tite 1986: 59, but see discussion below). Interestingly, the use of grog from broken crucibles or burned bricks is recommended in the *Probierbüchlein* just as a way of saving sand (Sisco and Smith 1949: 111). Ercker notes that either grog or crushed pebblestone may be used as temper, but the latter "serves as a binder in the fire, and the greater the heat, the faster becomes the bond. Scorifiers made in this way come out of the molds clean and intact." (Sisco and Smith 1951: 24-25)

It appears that all the scorifiers would have shown a sufficient thermal refractoriness, given their high alumina content (26-33 wt%), together with the low soda, magnesia, lime (each $\leq$1 wt%) and potash (typically $\leq$2 wt%). Graphite-tempered scorifiers may be assumed to be the most refractory ones, but only so long as graphite remained stable. The scorifier OB 269, with slightly lower alumina and higher magnesia and lime concentrations, appears as the least refractory of the group (Table 4, fabric C). However, it is not thermally distorted, which may be related to the fact that the highest temperature range was not kept for a long period, as also indicated by the clean interface between the slag (containing as much as 87.6 wt% lead oxide) and the ceramic body, with very little formation of lead silicate (fig. 75, right; see discussion below).

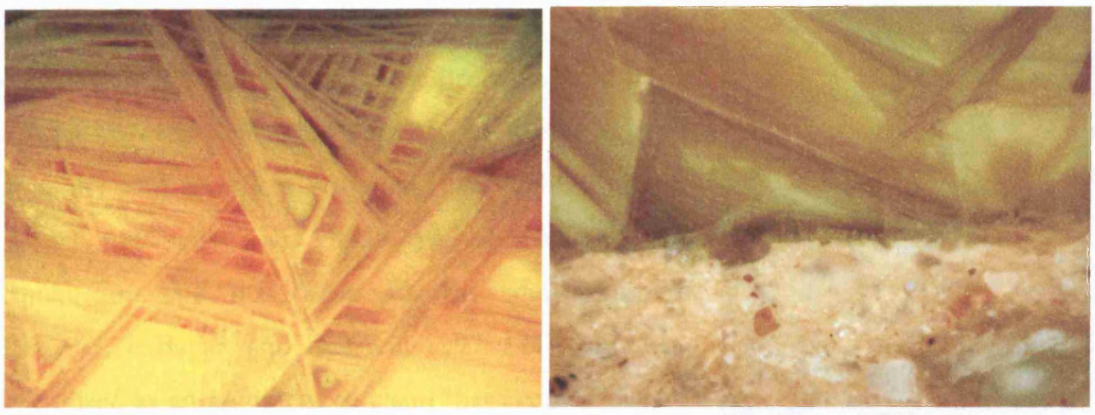


Figure 75. Two photomicrographs of the cross-section of scorifier OB 269. Left, detail of the big litharge crystals formed within the slag as a result of a slow cooling rate. Right, detail of the interface between slag and ceramic, showing little interaction (both OB 269/s1, XPL, 100x, long axis represents ~1 mm).

Generally, as with the triangular crucibles, thermal refractoriness was not a major technical impediment for the scorifiers. All the samples analysed, irrespective of their specific conditions of use, show different stages from initial to intermediate vitrification, normally increasing towards the inner surface, but none of them appears drastically distorted by heat (fig. 76). Chemical refractoriness, on the contrary, may have been a more severe technical constraint. As Glauber (1651: 320) would write, "I never yet saw any earth which could hold litharge in the fire". Many scorifiers exhibit different extents of penetration by the lead oxide-dominated slag. It is not uncommon to find samples where the slag has slipped through the whole body, reacting with the silica of the fabric and forming a glassy lead silicate that exudes at the base (fig. 77). This would have meant a major hindrance for the performance of the vessels, and poses some questions as to the suitability of the siliceous scorifiers for their primary uses.

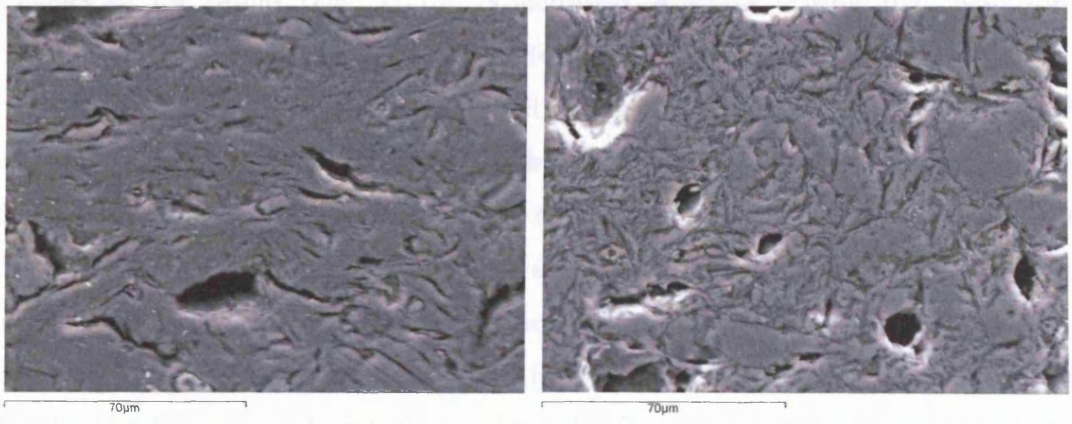


Figure 76. SE micrographs at the same magnification of two used scorifiers, showing initial (left) and intermediate (right) vitrification stages. None of them seems to be at risk of terminal distortion (left, OB 279/s1; right, OB 282/s1; both at 800x).

Figure 77. Top and bottom view of one of the several crucibles where the PbO-rich slag has penetrated through the whole siliceous ceramic body (OB 236).

In spite of the aggressiveness of pure lead oxide towards silica, this corrosion process would have a limit. Recent analytical research on archaeological crucibles and scorifiers has noted lead-rich slag with up to ~80 wt% lead oxide that seems to reach equilibrium and no longer absorbs silica (Rehren 1996a: 137; Bayley and Eckstein 1997: 111). This has been used as an argument to claim that ceramic dishes could be used for cupellation. Siliceous ceramics would not be as easy to use for this purpose as ash-based cupels, in that the latter readily absorb the lead oxide as it separates from the metallic *regulus* (see below, 6.4.1), whereas the former simply 'holds' the charge, and the lead oxide has to be skimmed off the surface so that the oxidation of the metal bullion can proceed, as noted, for example, by Agricola (Hoover and Hoover 1950: 239). Furthermore, even if the dissolving of silica would stop when the equilibrium was reached, the formation of lead silicate would not necessarily be constrained to the surface of the dish. Some of the samples from Oberstockstall show that slag easily penetrated through cracks so that corrosion took place further into the ceramic, which would challenge the stability of the vessel and the accuracy of the results (figs. 77-79). Nonetheless, it seems feasible that, with some experience, scorifiers could indeed be used for initial refining noble metals.

The PbO-SiO_2 system shows several phases in equilibrium with ~70 to 100 wt% lead oxide between 800 °C and 1000 °C (Jak *et al.* 1997; Jak *et al.* 1999). Accordingly, the slag layers within several of the scorifiers from Oberstockstall, with lead oxide levels above 70 wt%, would still be aggressive against silica at temperatures usual for scorification (fig. 78). This might be another reason explaining why those clays with a lower silica to alumina ratio were reserved for crucibles and scorifiers. In cross-section, the slag can be seen preferentially attacking the free silica within the ceramic matrix, whilst the quartz grains, even though composed of pure silica, have a low surface relative to their volume and thus offered more resistance to corrosion (fig. 79). Therefore, although the bulk chemical composition of a quartz-tempered fabric would

appear richer in silica and hence less resistant to corrosion by lead oxide, the form in which this silica is present would make a significant difference. This stresses the pertinence of analysing the composition of the ceramic matrix separate from bigger inclusions (see section 5.5).

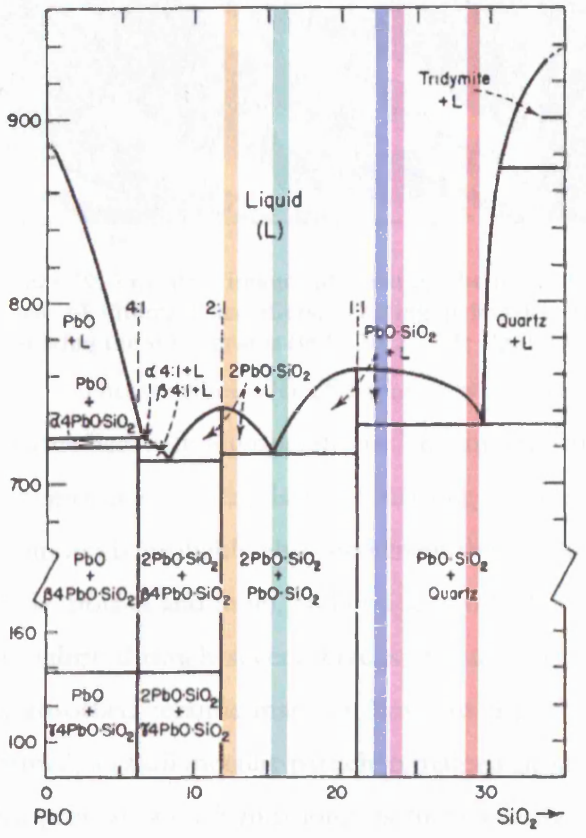


Figure 78. PbO-SiO_2 phase equilibrium diagram. The vertical lines plot the PbO concentrations of the slag layers within (from left to right) scorifiers OB 269, OB 282, OB 277, OB 276 and OB n006. Note that, for temperatures above 800 °C, the system would still be aggressive against silica in all cases (after Levin *et al.* 1973, fig. 284, modified).

Bayley and Eckstein (1997) have suggested that, before the 16th century, scorification and cupellation may have constituted a continuous process of oxidation on successive siliceous trays that were used as long as they were stable. For the scorifiers from Oberstockstall, the relatively big size of the metallic *reguli*, as documented in the depression left within the slag in some dishes (fig. 65, p. 143), suggests that the scorifiers were used only for an intermediate oxidation, while the final refining would be achieved in the bone ash cupels (see below, section 6.4). Such a practice appears documented in the *Probierbüchlein* (Sisco and Smith 1949: 110). In this sense, the use of the scorifiers could be seen as an intermediate step primarily aimed at reducing the sample size by oxidising part of the lead bullion, before moving on to the more sophisticated and efficient – yet more difficult to obtain – bone ash cupel (cf. Bayley and Eckstein 1997). Alternatively, the scorification stage might be aimed at melting a sample with a flux in oxidising conditions, to ensure the separation of the gangue before cupellation[22].

[22] Bayley (2003: 6) has suggested that scorifiers may have been used for cupellation when full quantification was not necessary. However, the 'losses' in a scorifier might have been considered equivalent to those unavoidable in the large scale counterpart, hence justifying the use of scorifiers for quantitative operations (see below).

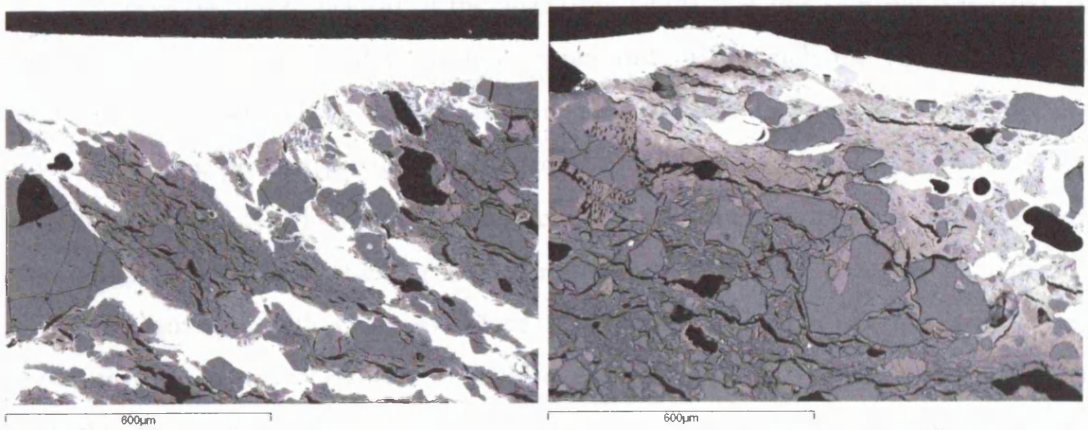


Figure 79. Two BSE images illustrating the penetration of lead oxide-rich slag (bright) into the fabric of different scorifiers, reacting primarily with the ceramic matrix and, more slowly, dissolving the silica grains (left, OB 277/s1; right, OB276/s1; both at 100x).

The scorifier OB 282 may serve as an example of the use of these dishes for intermediate refining operations. The internal surface of this sample appears covered by a homogeneous slag layer, consisting largely of lead oxide (84.5 wt%) containing compounds probably absorbed from the ceramic material (namely, silica, alumina, iron oxide, potash and lime), and copper oxide (0.4 wt%). This slag appears penetrating into the fabric through several cracks, where its composition reflects a higher contribution by absorbed ceramic material. Towards the edge of the central space where the bullion formed, a small metallic particle remained sitting on top of this slag layer. The matrix of this prill, about 1.5 mm long, is (now mostly oxidised) lead containing relatively high concentrations of silver (4.6 wt%) and minor amounts of copper (0.5%), enriched on the surface in arsenic, calcium, phosphorus, copper, sodium and chlorine. Many dendritic crystals of copper and silver can be observed within the metallic lead, sometimes with traces of aluminium and nickel.

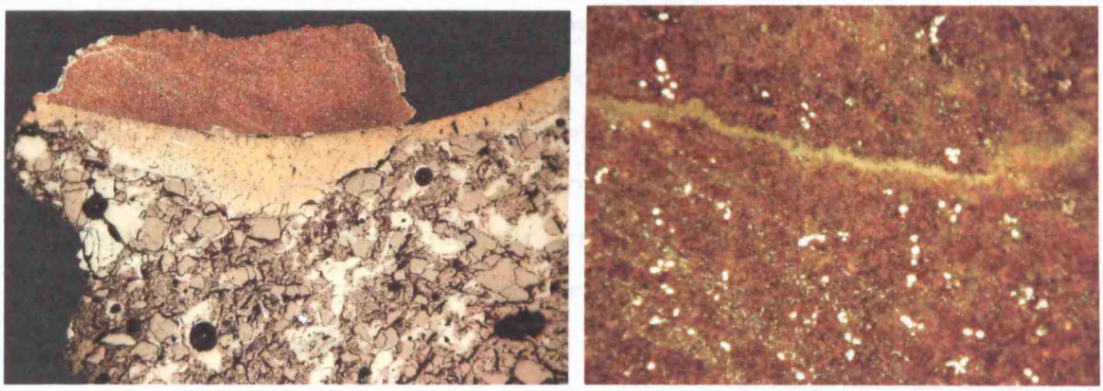


Figure 80. Left, cross-section of a scorifier with lead silicate slag (yellow) penetrating into the fabric, and an argentiferous lead droplet sitting on the inner surface (top). Right, detail of the lead droplet with bright dendrites of silver and copper (OB 282/s1, PL; left, 50x, width of particle is ~1 mm; right, 500x, long axis represents ~100 µm).

A possible interpretation of the analytical data is that this scorifier was used for the initial concentration of silver from a copper and/or lead-rich alloy, perhaps adding more metallic lead. This would constitute an intermediate stage in advance of cupellation. In a process possibly similar to that documented here, Agricola (Hoover and Hoover 1950: 249ff) described a simple method of assaying an alloy of copper and silver, which started by melting the sample in a triangular crucible in reducing conditions, covered with charcoal. Once the silver-rich copper was molten, it was poured in water to make granules, or cooled and cut into small pieces by other means. Normally these would go straight into the cupel. However, in some cases, he recommends:

> "If the copper be such that the silver can only be separated from it with difficulty, then before it is tested with fire in the cupel, lead should first be put into the scorifier, and then the copper should be added with a moderate quantity of melted salt, both that lead may absorb the copper and that the copper may be cleansed of the dross which abounds in it." (Hoover and Hoover 1950: 251)

There are more cases where the slag adhering to the scorifiers is a relatively clean lead silicate containing only some impurities absorbed from the ceramic and varying amounts of copper oxide, sometimes with traces of silver, likely remains of similar scorification processes.

Another sample supporting the idea of the scorifiers used for an intermediate oxidation stage is OB 284 (fig. 81). The thick (~2 mm) layer of slag adhering to the rim of this plate is mostly composed of oxides of antimony (45 wt%) and lead (34 wt%), together with iron oxide (10 wt%). It contains several relatively big (≤0.1 mm) droplets of metallic antimony and lead-antimony alloys, sometimes with traces of arsenic and copper (each ≤2 wt%), as well as molten ceramic components, and calcium and iron oxides that appear recrystallising. In all likelihood, this represents the scorification stage of an ore – possibly a fahlore – previously processed in a triangular crucible, such as those outlined in the previous section: the elements present are the same, but there are no raw grains of gangue or fluxes, and metallic elements are more abundant. Cupellation would probably be the next step.

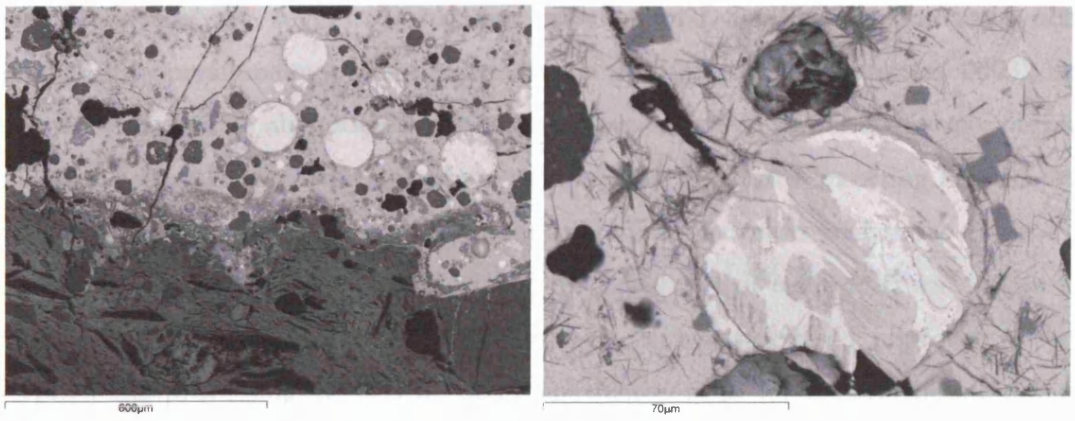


Figure 81. Left, interface between the graphitic ceramic (bottom) and the slag within scorifier OB 284, which shows big metal droplets (light globules) and a heavy absorption of ceramic material (dark phases within slag). Right, detail of the slag, with a big lead/antimony prill (centre), some smaller antimony droplets (light), cubic iron oxides and star-like crystals rich in silica, lime, iron oxide, alumina and magnesia (OB 284/s1, BSE, left at 50x, right at 800x). See Appendix 2 for analytical data.

In sum, scorification could render the samples more suitable for cupellation, while reducing their volume. The ceramic dishes would probably have to be watched carefully in order to make sure that the process was stopped before they became unstable, but in any case they would offer a simple, 'good enough' resort for this intermediate step. It should be emphasised, however, that most of the slag found within scorifiers bears some metallic droplets, possibly owing to the speed of the process and the need for skimming the lead oxide off the centre of the scorifier for the oxidation to proceed. An interesting question is whether those conducting the scorification were aware of these losses and accepted them as unavoidable. Lazarus Ercker warns that the assay with a scorification stage "gives a reliable indication of how the smelting operation should be planned and conducted; but it does not enable you to find the correct silver content of the ore." (Sisco and Smith 1951: 41). For a more accurate result, big bone ash cupels should be used for the entire process, skipping the scorification stage. However, he adds the very relevant observation that, in the large-scale smelting of ores, the forming slag will also retain some silver. Accordingly, an assay with some metal losses during scorification might represent a more realistic reproduction of the large-scale counterpart (Sisco and Smith 1951: 42).

6.3.3. The choice of ceramic material

As stated above, no significant correlation was identified for the scorifiers between fabric and formal types, or between these and different traces of use. Although

preliminary analyses suggested that graphite-tempered scorifiers might have been reserved for operations requiring higher temperatures (Martinón-Torres *et al.* 2003), this hypothesis had to be abandoned as the sample expanded. At present, the only distinct feature seems to be the use of graphitic scorifiers for processes involving the removal from the furnace while hot, but the number of graphitic scorifiers analysed is too low to allow definite conclusions. In view of this, the variability of ceramic fabrics might be interpreted as an indication of a comparatively less standardised approach to the selection and processing of clays for scorifiers than noticed for the crucibles, although still within a relatively narrow range.

The identity of the ceramic fabrics between both graphitic crucibles and scorifiers suggests a common provenance (see section 7.5). As regards the second ceramic type of scorifiers, the very similar chemical composition of the matrix and the analogous formal appearance of the dishes make it reasonable to conclude that they also came from the same source. The slightly different composition of their matrices could be related to the exploitation of several clay deposits within the same region, in all cases characterised by the low alkalis and earth alkalis content and a relatively low silica to alumina ratio. Some scorifiers formally analogous to the deeper type described above have been found in Saalfelden (Pittioni 1978) and Steiermark (Sperl 1996), two other Austrian sites. In the lack of analyses of their fabrics, it seems reasonable to infer the existence of a relatively wide trade of specialised technical ceramics, which would include at least crucibles and scorifiers (see section 7.5).

Figure 82. Example of a scorifier with little traces of use. Note the dark grey surface, akin to that of the crucibles.

Assuming that most scorifiers came from the same producers as the crucibles, it would be interesting to know whether they shared the black lustre interpreted as a quality signifier or 'trade mark' for the crucibles. Those scorifiers with less substantial traces of use show a suggestive grey colour all over the surface, perhaps a remnant of the original appearance, similar to that of the crucibles (fig. 82). However, the lack of truly unused samples leaves this hypothesis untested. Furthermore, another question would be why most of the crucibles were stamped by the potters, whereas none of the scorifiers shows house marks.

The only scorifier for which a different provenance might be hypothesised is OB 269, with distinctive chemical composition and mineral inclusions (see above, fabric C). This is the biggest and the least refractory of those analysed. An interesting feature of this sample is that the slag appears slightly edging over the central space where a relatively big (~60 mm) metal button must have been sitting (fig. 83). As a consequence, the plate had to be broken in order to retrieve the metal, and the plate was necessarily discarded after use. This was probably the fate of most scorifiers – at least those used for scorification –, given the generally strong distortion of the ceramic by the hot slag, as well as the risk of contamination if quantitative inferences were to be made on the results of the assay made on a re-used dish.

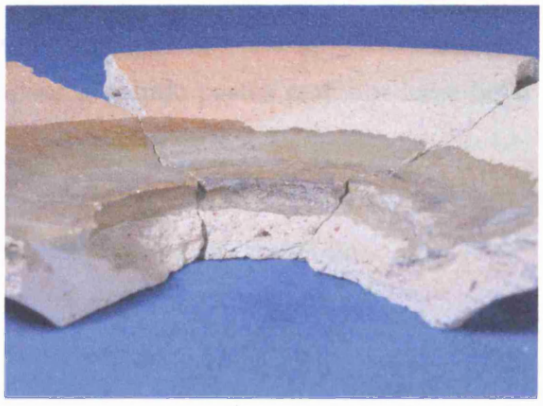


Figure 83. View of scorifier OB 269, showing the concave central space probably left by the metal button.

6.3.4. The manufacture and performance of the scorifiers: a summary

The scorifiers recovered in Oberstockstall were made with a relatively narrow range of clays which were processed in different ways before being modelled on a potter's wheel. However, it is likely that most, if not all, were manufactured by the same producers as the crucibles, who possibly smoked them black too. One main fabric type is characterised by the presence of graphite inclusions, while the other one contains grog temper. For both types, the ceramic matrix shows a remarkably high alumina content.

The analytical data indicate that most scorifiers were used for transitional operations within an assaying or refining sequence. They were frequently used for the oxidation of a lead-dominated bullion in order to concentrate the noble metals in a smaller sample prior to cupellation in ash-based cupels. Other high-temperature reactions are also documented, though.

Scorification reactions must have been carried out quickly, given that the lead oxide-rich slag was very aggressive against the free silica within the ceramic matrix, and that graphite inclusions often burned out leaving large voids.

The use of a thick, alumina-rich ceramic, together with the presence of quartz inclusions that functioned as 'buffers' for corrosion, enhanced the technical proficiency of the vessels, although this did not preclude some of them from failing. However, the question whether this was a deliberate choice can only be addressed after a comparative view of the whole assemblage (see section 6.5). Finally, there is no clear difference in the utilisation of graphitic and non-graphitic scorifiers. This accords well with the above hypothesis that the peculiar material properties of graphitic pastes may not have been well realised at this time.

6.4. The bone ash cupels

The chemical foundations, as well as the history and archaeology of cupellation, have been addressed in length elsewhere (Bayley 1991; cf. Bachmann 1993; Rehren and Klappauf 1995; Bayley and Eckstein 1997; Pernicka *et al.* 1998; Ramage and Craddock 2000; Rehren and Eckstein 2002), therefore just a brief introduction is given here. Cupellation is a metallurgical operation aimed at retrieving the noble metals contained in a larger metal matrix or 'bullion', normally dominated by lead. The metal is melted under oxidising conditions so that the lead is oxidised to litharge, which acts in two ways (Bayley 1991: 125): it oxidises the other base metals possibly present, and it reacts with these metal oxides, forming fusible compounds. As a result, the noble metals, which do not react with oxygen or lead oxide, separate from the melt as a button or *regulus*.

The ideal medium for cupellation is a porous, non-siliceous substrate, which does not react with the oxidised metals but absorbs them by capillary action, while the *regulus*, sustained by its higher surface tension, settles on top (Rehren and Klappauf 1995). This technique can be traced back to the Early Bronze Age, where it seems that lime-rich clay was specifically selected to coat the cupellation hearth (Pernicka *et al.* 1998). Numerous 'litharge cakes' have been recovered in a variety of later archaeological contexts (Bachmann 1977; Bayley 1991; Bayley 1992; Keesmann 1993; Rehren and Klappauf 1995; Bayley and Eckstein 1997; Rehren and Kraus 1999; Ramage and Craddock 2000). These formed on the upper part of cupellation hearths as the carbonate- (clay marl or shell) or phosphate- (bone ash) based matrix absorbed the litharge and other metal oxides during the process. Siliceous matrices were deliberately avoided, probably as a result of an ineffective performance, given that silica reacts with litharge to form viscous lead silicate glasses that would choke the porosities and hinder the absorption process.

The small-scale counterpart of cupellation is carried out in the thick, ash-based vessels known as cupels. The cupel is probably the most specialised vessel type related to fire assay, as it could serve no other purpose than cupellation. It should be emphasised, however, that cupellation may be related to either production, or recycling or assaying (Bayley and Eckstein 1997), hence cupels may be found in mining contexts as much as in mints, jewellers' workshops or chymical laboratories. This stresses the need to investigate the wider context in order to interpret archaeological remains (see chapter 2, and Martinón-Torres and Rehren 2004a).

Cupellation in ash-based cupels is described in alchemical sources as early as the 8th century AD (Hoover and Hoover 1950: 465-466, n. 26; Halleux 1986; Szabadváry 1992; Newman 2000; Newman and Principe 2002: 35-49). Nonetheless, the regular use of these vessels outside the alchemical domain does not appear widespread until several centuries later – which may constitute an interesting case of technology transfer. As noted in the previous section, ceramic scorifiers could have been used for cupellation during much of the Middle Ages. In the 12th century, Theophilus (Hawthorne and Smith 1979: 96-97) describes an ash-lined earthenware dish, a tool that marks the transition between scorifier and cupel, and has been matched by a roughly contemporary archaeological find (Rehren and Eckstein 2002). Since late medieval times, bone ash cupels appear in increasing numbers and contexts across Europe and beyond (Bayley 1988; Jeanjacquot 1993; McLees 1996; Rehren 1996a; Schifer 1998; Unglik 2000; Lopes *et al.* 2000; Saunders 2001; Bayley 2003).

6.4.1. Formal and material properties

The standard shape of a cupel is that of a truncated cone with a shallow concavity on the top (wider) side, where the sample to be cupelled would be placed. This space is surrounded by a neat rim. Of the approximately one hundred cupels recovered in Oberstockstall, only one (OB 957) had not been used, this being white in colour. In this sample, it can be noticed that the top surface was lined with a finer, more smoothly packed, material. This is in accordance to a recommendation made in contemporary treatises, which Ercker even labels as "an essential requirement for cupels" (Sisco and Smith 1951: 31). The rest of the cupels are notably heavier and show colours spanning from buff to dark grey or brown.

Four size ranges were tentatively identified by von Osten (1998: 50): the first type has a biggest diameter of 30-35 mm and a height of 15-20 mm; the second is 40-45 mm in diameter and 20-30 mm high; the third type is 55-65 mm in diameter and 30 mm high, and those of the largest size have a biggest diameter of 85-100 mm and a height of 45 mm (fig. 84). Most of the samples fall within the smaller ranges. Lazarus Ercker (Sisco and Smith 1951: 33) and, following him, Libavius (Rex 1964: II, 13), define four size categories for the cupels with their respective uses. At present, however, given the relatively small sample analysed, it is not possible to ascertain any correlation between the sizes and uses of the cupels from Oberstockstall (fig. 84).

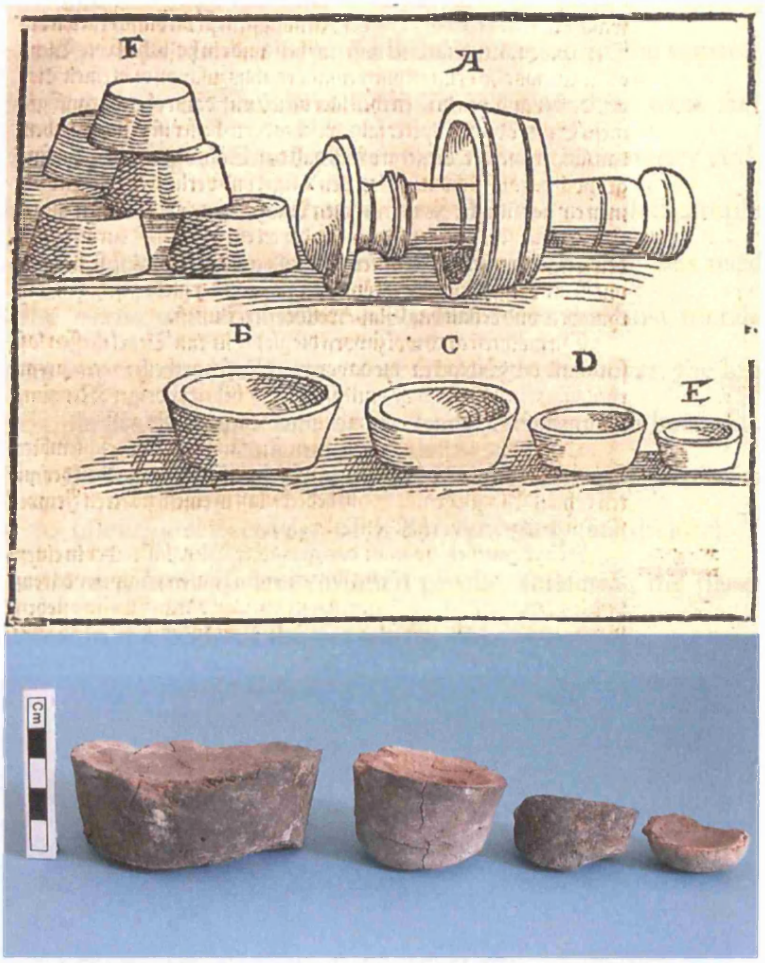


Figure 84. Top, bone ash cupels and the mould for making them as represented by Ercker (Sisco and Smith 1951: 33, fig. 6). Bottom, some of the one hundred cupels recovered in Oberstockstall.

The conical shape of the cupels would make it easier to release them from the mould when manufactured (fig. 84). The substantial thickness of the bodies is explained by the need of a porous medium to absorb the lead oxide and other base metals as they oxidised. According to Unglik (2000: 75), the maximum amount of litharge that a pure bone ash cupel within the second size range could hold without leakage would be that produced from a 30 to 45 grams lead charge. In the

Figure 85. Example of a cupel whose whole body is soaked in lead oxide.

assemblage from Oberstockstall, the only unused cupel preserved weights 26 g, whereas some o fthe used ones reach above 140 g. With the exception of the unused cupel, those cupels that preserved their original shape did so because they were fully soaked in litharge, which aupon cooling solidified, thereby conferring consistency and durability to the vessel (fig. 85). For these cases, we may assume that the cupelled sample was too big for the cupel, as the absorption capability of the porous substrate was used to its limit, possibly before the *regulus* was completely separated from the host metal. This would probably lead to an imperfect cupellation. Most frequently, however, the litharge did not soak the cupel completely, leaving some of the bone ash near the base unconsolidated. This 'clean' part of the vessel is no longer present, either through erosion of the friable material, or due to intentional recovery of it for recycling (see below). The bases of these cupels thus show a more or less rounded profile, reflecting the penetration front of the lead oxide within the body of the vessel (fig. 86).

Figure 86. Some upturned cupels, most of them showing rounded shapes.

6.4.2. Used cupels and performance

A detailed analytical investigation of some of these cupels, involving X-ray diffraction (XRD), scanning electron microscopy (SEM) and inductively coupled plasma – optical emission spectrometry (ICP-OES), was conducted by Thilo Rehren (1998; see also Martinón-Torres and Rehren 2004b). Apart from offering a pilot approach to the use of these vessels and posing some queries for future work, this study presented also a preliminary assessment of the cupels' manufacture and performance. Knowing that the ratio of P_2O_5 to CaO in bone apatite is about 1:1.2, the likely bone ash component of the cupels could be subtracted from their bulk compositions (Table 5). This showed the used cupels to be made of about two thirds finely crushed bone ash and one third calcareous or dolomitic clay mixed in as a binding agent. Among nine cupels analysed,

this clay component indicated the use of two different types: one being richer in magnesia and lime than the other, which in turn is higher in potash and silica.

The relatively high amount of clay relative to bone ash exceeds clearly what 16th-century authors such as Agricola (Hoover and Hoover 1950: 228-230), Biringuccio (Smith and Gnudi 1990: 137-139) and Ercker (Sisco and Smith 1951: 26-33) suggest; they generally recommend using pure bone ash, mixed with egg white or water as a binder. Alternatively, they accept the use of wood ashes or a mixture of both, although noting that those made of pure bone ash perform better. Lazarus Ercker, even though discouraging this practice, mentions the possibility of using "a tenth part of good potter's clay," provided that "the clay itself is good and fire-resistant; otherwise it does the cupels more harm than good" (Sisco and Smith 1951: 29).

Microscopic examination of the used cupels demonstrated how the bone apatite becomes unstable in the presence of silica, reacting with the lead oxide to dissolve and recrystallise as complex calcium silica phosphates (Rehren and Eckstein 1996; Rehren 1998), and in doing so clogging up the porosity needed to mechanically absorb the liquid lead oxide. This is probably reflected in the significantly lower concentration of lead oxide in the first group of cupels (on average 64 wt%), which is richer in silica, compared to the second group (*ca.* 73 wt% PbO), which is richer in magnesia and lime (Table 5).

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	FeO	clay	bone	Bi+PbO
OB 936	2.1	4.6	10.9	68.6	6.6	2.4	5.1	11.2	15.4	64.2
OB 876h	1.9	5.3	11.3	68.3	7.2	3.5	4.3	12.2	18.0	60.5
OB 943	2.7	6.2	10.5	66.8	6.4	2.4	5.5	13.1	18.9	60.0
OB 917	2.6	8.8	10.7	70.3	4.5	2.8	2.2	8.2	19.1	65.1
OB 932	1.9	10.0	10.6	68.5	4.9	2.4	3.0	7.8	15.6	70.3
OB 928	2.6	11.6	12.0	64.0	3.5	7.7	5.6	4.7	11.8	69.8
OB 876d	1.5	16.8	8.7	70.0	1.5	3.2	1.4	5.8	11.1	74.3
OB 919a	2.1	17.6	8.9	62.0	3.0	6.0	3.9	5.6	8.8	74.9
OB 918b	1.0	18.4	11.3	60.9	1.2	5.9	6.0	6.7	7.9	72.4

Table 5. Reconstructing the raw materials of cupels from Oberstockstall, and their ability to absorb lead oxide. The first seven columns detail the theoretical clay composition (columns Na_2O to FeO), followed by the original combined weight percentage of these oxides in the used cupels (column 'clay'), the percentage of bone ash in them (column 'bone'), and the sum of bismuth and lead oxide found in the used cupels (last column). The theoretical clay composition was obtained by first reducing the total CaO content by 1.2 times the weight of P_2O_5 to account for bone ash, and normalising the light oxides including any remaining CaO to 100 wt%. The sum of any P_2O_5 and 1.2 times this weight in CaO was defined as the likely bone ash/apatite component. ICP-AES analyses by W. Steger, Deutsches Bergbau-Museum, Bochum (full details in Rehren 1998; table from Martinón-Torres and Rehren 2004b).

6.4.3. Making cupels – recycling cupels?

During a study visit to the *Museum Der Alchemist* in 2001, a lump was noticed of a white, flour-like powder (fig. 87). This appeared compressed in a roughly cylindrical shape with a wider belly, about 15 cm high and 10 cm in diameter, which suggested that it could constitute the content of a small wooden barrel that had decomposed. Suspecting that this might be raw material used for the manufacture of cupels, a sample was removed for further investigation.

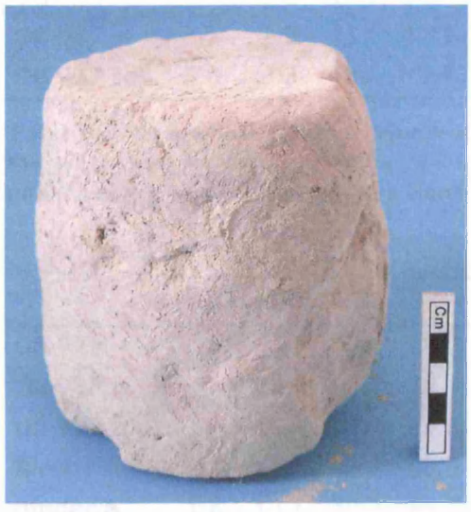


Figure 87. Lump of white powder recovered in Oberstockstall (OB 925).

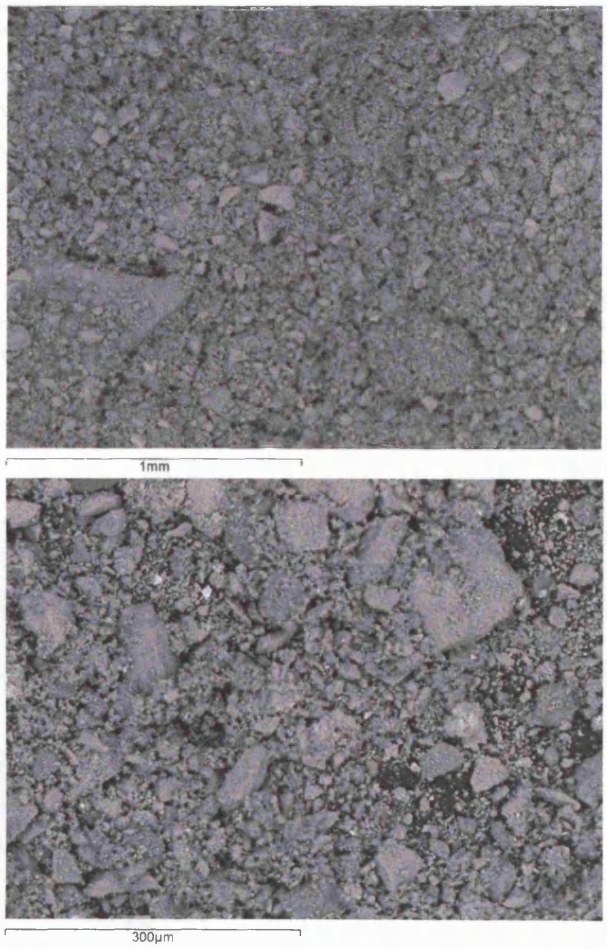


Figure 88. Two views of the white powder probably used as raw material for making cupels. The chemical composition of some of the bigger crystals is presented in Appendix 1 (OB 925, BSE, top at 60x, bottom at 200x).

SEM-EDS analyses revealed that the powder consisted of an agglomerate of very fine-grained bone ash and clay, containing small, angular calcium silica phosphates, all of them ≤100 µm in diameter (fig. 88). The bulk composition of this powder showed high phosphate and lime concentrations, indicative of bone ash (Table 6, top row). However, when the likely apatite component was neglected (as in Table 5) and the results normalised, it became noticeable that the lime in the remaining matter was significantly high (Table 7). In addition, the powder showed a substantial copper oxide content. Furthermore, many spongy particles were identified within this paste, most of them with copper oxide and lead oxide as main elements, but also containing phosphate

and lime, as well as varying amounts of iron, nickel, zinc, arsenic, bromine, silver, antimony and tin (fig. 89).

	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	MnO	FeO	CuO	ZnO	Ag	PbO
OB 925	1.9	3.3	11.8	26.4	3.7	0.4	1.1	44.8	-	4.6	2.2	-	≤0.2	-
OB 937	3.5	2.3	13.5	12.1	0.3	-	1.3	64.6	-	1.8	0.9	0.1	-	0.7
OB 937 top	5.0	3.3	18.6	9.7	0.3	-	1.3	59.7	0.3	1.8	-	-	-	-

Table 6. Average bulk chemical composition by SEM-EDS, normalised to 100 wt%, of: OB 925, the white powder interpreted as raw material for making cupels; OB 937, the body of the unused cupel; and OB 937 top, the fine top layer of the same unused cupel.

	Na_2O	MgO	Al_2O_3	SiO_2	SO_2	Cl	K_2O	CaO	MnO	FeO	CuO	ZnO	Ag	PbO
OB 925	-	4.5	7.8	28.0	8.8	0.9	2.6	31.1	-	10.9	5.2	-	≤0.2	-
OB 937	-	4.7	3.1	18.1	0.4	-	1.7	67.2	-	2.4	1.2	0.1	-	0.9
OB 937 top	-	6.4	4.2	23.6	0.4	-	1.7	61.1	0.4	2.3	-	-	-	-
Binder A	2.2	7.0	10.8	68.5	-	-	5.9	2.7	-	4.0	-	-	-	-
Binder B	1.8	16.1	10.2	64.2	-	-	2.3	5.7	-	4.2	-	-	-	-

Table 7. Theoretical composition of the clay or binder mixed with the bone ash for making cupels. The first three rows present data from the samples in Table 6 after neglecting the bone apatite component and normalising to 100 wt%. The bottom rows show the average composition of the two clay types identified in Table 5.

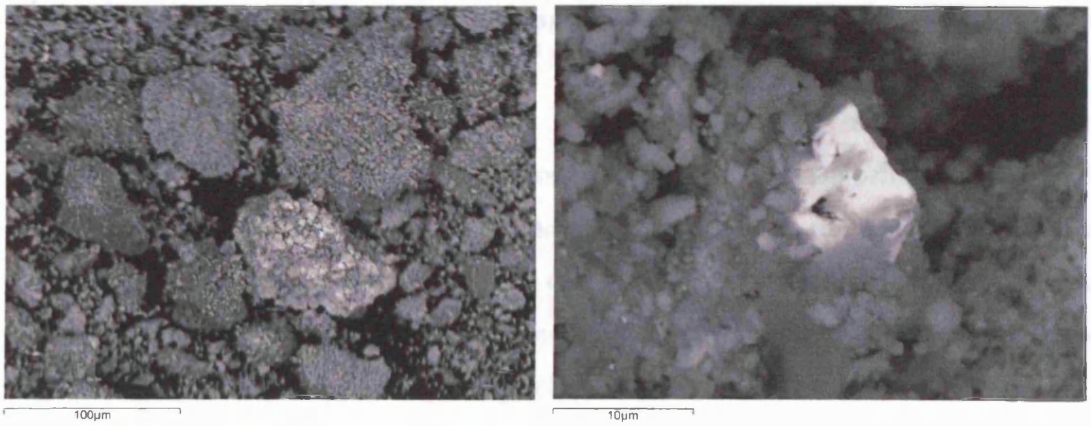


Figure 89. Detail of two of the bright particles of bone ash heavily impregnated with metal oxides, likely remains of cupellation processes (OB 925, BSE, left at 400x, right at 3200x).

In view of this, it could be proposed that this powder was indeed the raw material used for making cupels, but also that this material was recycled. The bone used for making cupels had to be boiled, calcined, crushed, ground, washed, sieved and dried, often repeating this process several times, before carefully pounding them in a mould and applying a finer top lining. Considering the amount of effort necessary for

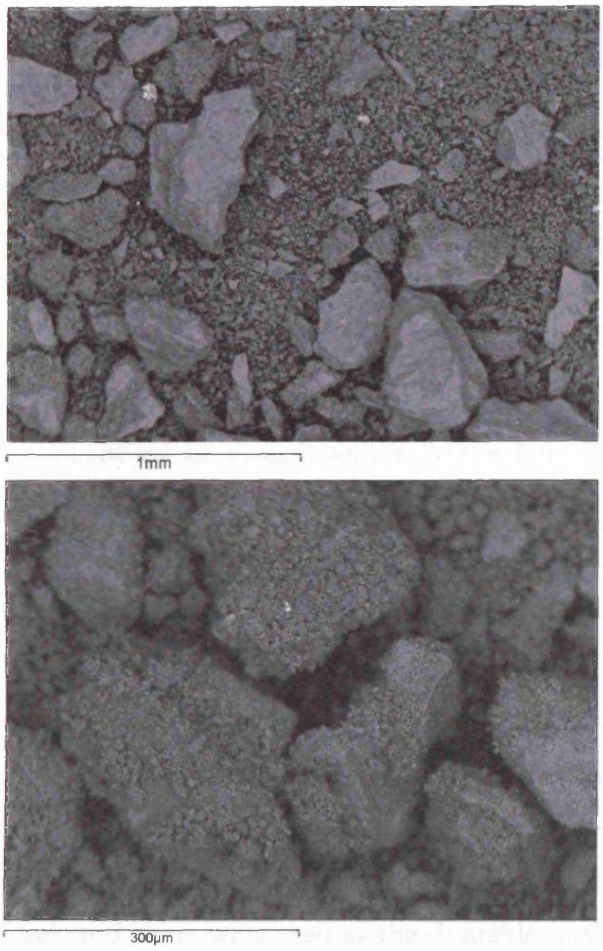


Figure 90. Two views of the body of the unused cupel. The chemical composition of some of the bigger crystals is presented in Appendix 1. Compare to images in fig. 88, taken to the same scales (OB 937, BSE, top at 60x, bottom at 200x).

preparing suitable clay-bonded bone ash for cupellation, it seems reasonable to accept that the artisans working in Oberstockstall scrapped off the relatively clean bottoms of used cupels, which could then be easily re-ground and recycled in the production of new vessels. This would explain the presence of some small spongy particles soaked with metal oxides – likely remains of previous cupellation processes, still preserving the characteristic texture of the bone ash –, as well as the overall copper oxide contamination of the powder.

Two questions regarding this material remain: on the one hand, the lack of significant traces of lead oxide, which one would expect to be the main contamination in recycled cupel material; on the other hand, the extremely high lime content, which does not match the composition of the used cupels. A possible explanation for the former problem might be the relatively small size of the sample analysed (<0.1 vol%), but no definite answer to this may be offered at the moment. The latter question shall also remain open, although some clarification arises from the analysis of the unused cupel (fig. 91).

Under the SEM, the body of the unused cupel OB 957 appears to be made of a substance very similar to that described above, although containing a larger proportion of relatively big particles (200-500 µm) (fig. 90). The bulk composition of this material roughly resembles that of the white powder, although it shows a lower iron oxide concentration and, more importantly, an even higher lime to phosphate ratio (5.3:1 compared to 1.6:1) (Table 6). It is tempting to attribute the high lime concentration in both cases to post-depositional contamination, namely the deposition of calcite from

the burial environment. In fact, if all lime and phosphate are neglected from the bulk compositions, and the results normalised, the resulting figures compare better to those from the used cupels – these, fully soaked in litharge, were less prone to the intake of post-depositional contaminants (Table 8). However, it should be noted that it was not possible to detect any calcite as such within the specimens analysed by SEM-EDS: calcium always appeared as a part of more complex compounds. Therefore, its precipitation would have occurred on a very small scale or, possibly, by absorption on the mineral phases of the matrix (cf. Freestone 2001: 621-622). Obviously, alternative explanations could be that, just as different clay types were used as binders, some batches of raw material may have been richer in lime or calcite, or even that calcite was added to the clay/bone mixture in some cases, perhaps experimenting[23]. It could even be suggested that, just as black and shiny were seen as the desirable qualities for a good crucible (section 6.2.3), the chymists in Oberstockstall might have been trying out different materials for cupellation, but all of them white and finely ground. The author cannot refrain from recalling some evocative verses by the alchemist Thomas Norton:

Figure 91. Top view of the unused cupel OB 957, showing the finer lining of the surface (photo Thilo Rehren).

> "*Colour* is the utmost thinge of a Body cleere,
> Cleere substance well termined is his matter heere;
> If Heate hath maistery in matter that is drye,
> *White* Colour is ever thereof certainely;
> As it appeareth in sight of brent Bones,
> And in making of all Lyme Stones." (Norton 1652: 55).

[23] The latter would, however, be detrimental for the performance of the cupels, since it would cause spitting as calcite decomposes to lime and carbon dioxide at temperatures above 840 °C.

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	MnO	FeO
OB 925	-	8.4	14.5	52.0	4.8	-	20.3
OB 937	-	15.7	10.3	60.3	5.7	-	8.0
OB 937 top	-	16.6	10.9	61.1	4.4	1.0	6.0
Binder A	2.2	7.1	11.0	69.6	6.0	-	4.1
Binder B	1.8	16.3	10.3	65.0	2.3	-	4.3

Table 8. Theoretical composition of the clay or binder as detailed in Table 7, after neglecting all the phosphate and lime content, as well as traces of use or post-depositional contamination such as sulphur, chlorine and heavy metal oxides, and normalising the resulting figures to 100 wt%.

Whatever the case, the resemblance between both samples, *i.e.* the powder in the white lump and the material of the unused cupel, supports the idea of a small-scale, possibly in-house, manufacture of cupels, which involved the recycling of the cleaner parts of used cupels. Interestingly, the unused cupel also contained spongy particles of metal oxides, likely contamination from previously used cupels (fig. 92).

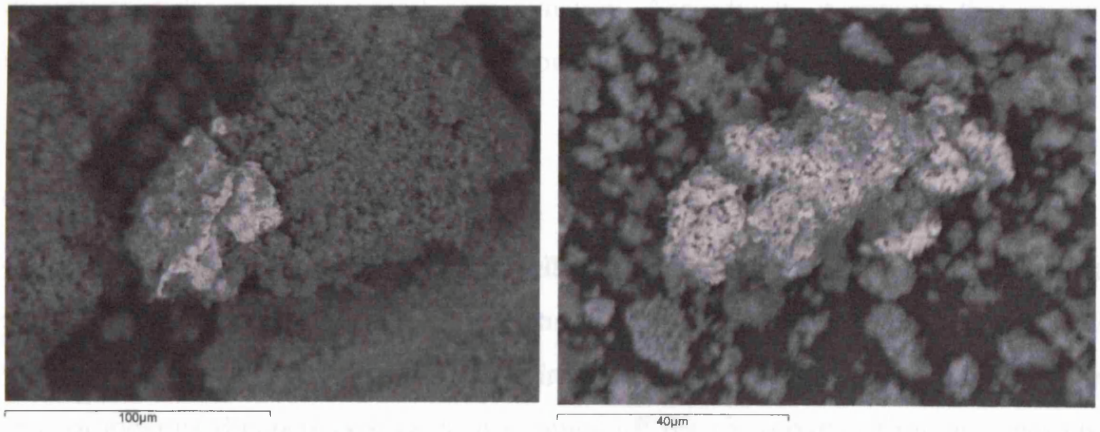


Figure 92. Detail of two of the spongy particles heavily impregnated with metal oxides and found within the body of the unused cupel, similar to those detected in the white powder, and likely indication that the cupel was made using recycled material (OB 937, BSE, left at 600x, right at 1200x).

Finally, a small fraction of the finer powder lining the top cavity of the unused cupel was also examined. Although the bulk composition of this specimen is rather similar to that of the body (Table 7), its average grain size is much smaller, and it appears more densely packed, indicating that more care was taken in its preparation (fig. 93). Moreover, in stark contrast with the other cupel raw materials analysed, this top lining does not contain heavy metal oxides of any sort, either as discrete particles or as a general contamination. Accordingly, it appears that freshly made clay-bonded bone ash was reserved for this surface lining.

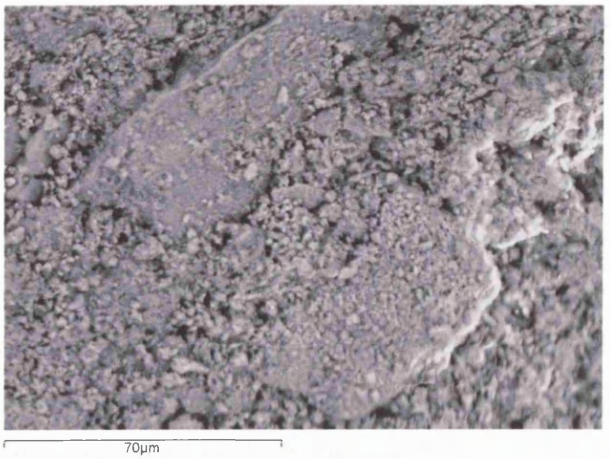


Figure 93. BSE photomicrograph of the top lining of the unused cupel. Note the fine-grained matrix and the small size of the crystals, in spite of the high magnification of the image. Compare to figs. 88 and 90 (OB 937, BSE, 800x).

6.4.4. Summary and further discussion

The scientific analyses of the cupellation remains from Oberstockstall suggest that cupels were manufactured from a mixture of finely crushed bone ash and clay, and that this material was recycled. This recycling could explain the rounded profile of the base of most used cupels as a result of scraping off the clean parts of the used vessels, contrary to the assumption that this shape is the result of post-depositional abrasion. This material would be collected for the manufacture of new cupels, to be carried out either by the chymists themselves or by somebody else.

Sixteenth-century written sources emphasise that the production of cupels is a very intricate process, with which most potters are not familiar. This explains why the chymists working in Oberstockstall, who got the bulk of their technical equipment from specialised producers, required a more autonomous strategy to ensure the supply of cupels. Mixing the bone ash with clay probably made it easier to mould the cupels, as it would confer more consistency to the paste. However, it also meant a decrease in the absorption ability of the vessel. The use of different clay types as binding agents for the bone ash, as indicated by the chemical analyses, further stresses the idea of a non-standardised manufacture.

The re-use of cupel material is not described in the written sources, and this constitutes the first archaeological evidence documenting such a practice. Normally, the used cupels would simply be put aside and stored for future processing because, as noted by Ercker "they contain some silver" and "can always be turned to profit by melting out the silver they contain" (Sisco and Smith 1951: 78). The recycling of pure bone ash in itself would not necessarily mean any compromise in the performance of the new vessels; rather, it could be seen as an intelligent routine. Moreover, the use of freshly made material for the top concavity shows a concern with minimising contamination. However, as pointed out above, the cupels from Oberstockstall were bonded with a substantial proportion of clay, which favoured the development of impermeable calcium silica phosphates under high temperatures. As the proportion of these crystals would increase with successive heatings over repeated recycling events, the absorption capability of the matrix would progressively decrease.

6.5. Comparative material from the Oberstockstall assemblage

In order to place the high-temperature technical ceramics in a wider perspective, the first step will be comparing them to other artefacts found within the same laboratory assemblage. While it offers a more comprehensive view of the laboratory equipment, this comparison shall advance the assessment of the technical standard and degree of specialisation of the different wares, as well as highlighting the choices made by the potters and chymists who were making, trading and using them.

A number of comparative samples were selected and mounted for analysis, trying to cover the whole spectrum of fabrics represented in the collection (see section 6.1). These include a green-glazed still head, two different transparent-glazed types, an unglazed cucurbit, an unglazed funnel, and two fragments of black containers (one of them stamped). The analytical results for these specimens will be outlined first, followed by a more inclusive discussion.

6.5.1. Characterisation

a) Distillation and sublimation equipment

The sample OB n004[24] is a fragment of one of at least six instruments of its kind recovered in Oberstockstall. These are internally transparent-glazed containers with an open profile, ~120 mm high and 200 mm in diameter. The most characteristic feature of these vessels is an internal grid or 'stand' at the upper third, consisting of two perpendicular ceramic coils that form a horizontal cross, supported by a central pillar. In some cases, the body bears four equidistant perforations near the rim (fig. 94). Although the function of these instruments remains unclear, it has been related to sandbath distillation,

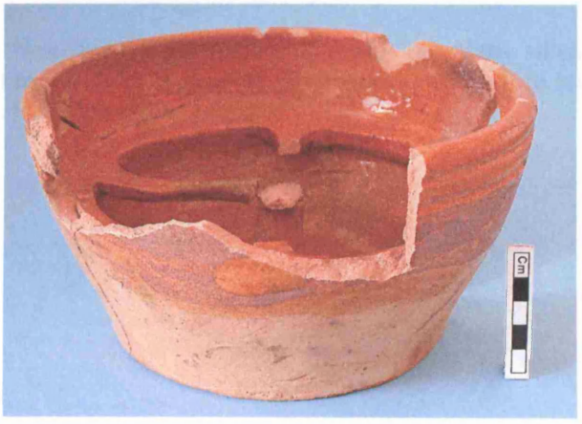


Figure 94. Instrument possibly used as a stand for a phial in sandbath distillation (OB 85). Scalebar is 5 cm.

[24] The specimen for analysis was taken from a bag, labelled with three find numbers (F.N. 16/3, F.N. 38 and F.N. 74) and containing several sherds without identification numbers, but its characteristic shape clearly allows the attribution of the selected sample to the instrument type discussed.

where they could function as stands for the phials, and as a means of water cooling (Soukup and Mayer 1997: 125-129).

The fabric of this apparatus is very similar to that of the non-graphitic triangular crucibles. The chemical composition of the matrix clearly falls within the same range (Table 9), while the size, shape and identity of the mineral inclusions are the same. As in these crucibles and the scorifiers, these also mostly quartz, together with some feldspar, rare iron oxide, hornblende, mica and tiny ilmenite, rutile and zircon particles. The colour of the paste is bright orange as a result of an oxidising firing, in contrast to the grey, reduced-fired, crucible fabrics. It shows only initial stages of vitrification. The glaze consists of a ~100 μm thick homogeneous layer of lead silicate containing minor amounts of alumina and iron oxide, together with traces of potash and lime (fig. 95; Table 10).

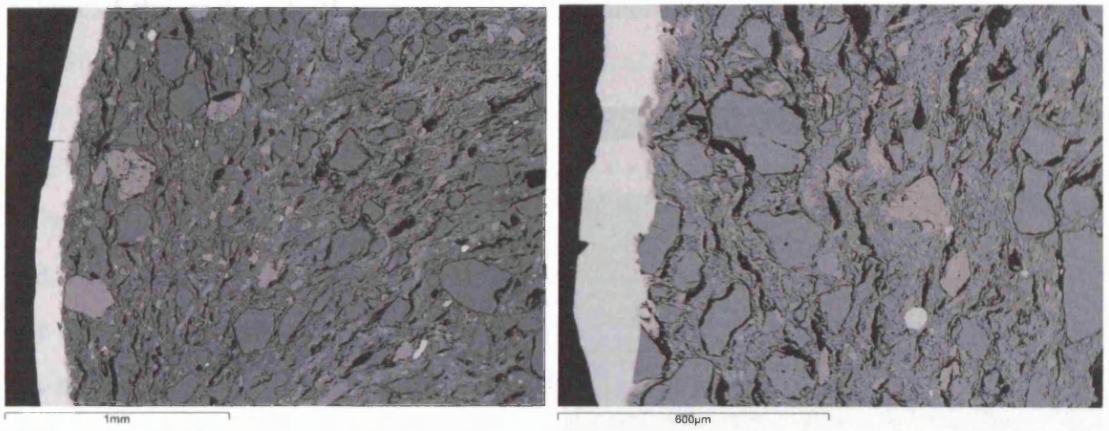


Figure 95. Two views of the fabric of the 'sandbath' instrument, showing abundant silicate inclusions, most of them quartz (dark grey) in a matrix with little vitrification, and a thin lead glaze layer on the surface (left) (OB n004/s1, BSE, left at 50x, right at 100x).

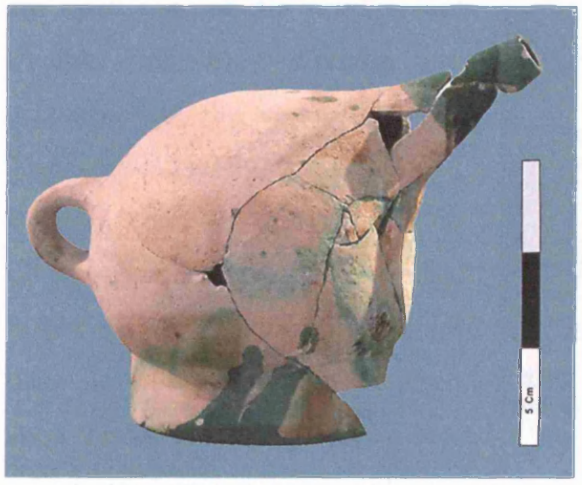


Figure 96. Green-glazed sublimation capital. The sample was taken from the base rim (OB 17). Scalebar is 15 cm.

The rest of the distillation and sublimation equipment analysed stand out because of the lower alumina and iron oxide concentrations in their ceramic matrices (Table 9). The instrument OB 17 is an alembic or capital used for sublimation. It has a globular shape, 150 mm high, with a vertical handle and a spout on top through which water or other materials could be added, while it served as an exit for vapour. The bottom part is open and has a rim that would fit in, and be luted to, the mouth of a cucurbit. The colour of the paste is buff to orange, but the inner surface is covered by a green glaze (fig. 96).

The fabric of this capital shows very little vitrification. It contains about 30 vol% silicate inclusions, subangular and with a moderate degree of sphericity, not very well sorted and reaching up to 0.5 mm in diameter. Most of these are quartz fragments – the biggest of which appear shattered –, although some feldspar is present, as well as rare amphibole, ilmenite, rutile and zircon. The ~200 μm thick glaze covering the inner surface is a lead silicate containing ~4.5 wt% copper oxide (fig. 97; Table 10).

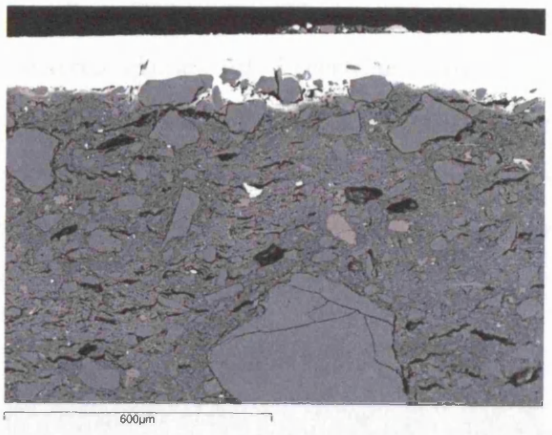


Figure 97. Image of the fabric of the capital. Note the cracks in the biggest quartz inclusion (dark grey), the glaze layer (bright), and the traces of the lute used to seal this capital to a cucurbit (top) (OB 17/s1, BSE, 100x).

Given its fragmentary condition, the next piece of distillation equipment analysed (OB n002) might be attributed to either a cucurbit (the pear-shaped container for the substance to be distilled, which would be covered by the capital) or a receiver (a similarly shaped vessel, connected to the spout of the capital to collect the distilled spirits). Both types are optically very similar in their fabric and shape, the main difference being the diameter of their rims – normally larger in the cucurbit, to match

the open base of the alembic, and smaller in the receiver, to accommodate the spout (fig. 98). The approximately 25 vessels of both types recovered in Oberstockstall have an average height of 300 mm and a maximum diameter of 180 mm. They often show traces of a thin layer of lute coating the outer surfaces, indicating a practice advised in contemporary treatises (e.g. Smith and Gnudi 1990: 196-197).

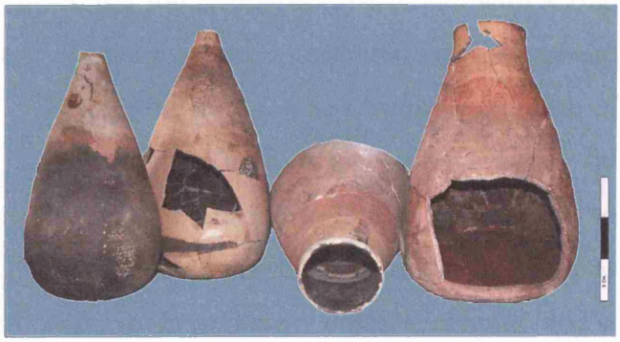


Figure 98. Some receivers (left) and cucurbits (right) from Oberstockstall. Scalebar is 15 cm.

The characterisation of the fabric of this instrument matches exactly that of the sublimation capital, except that the chemical composition of the matrix shows lower alumina levels and, conversely, a higher potash content (fig. 99, left; Table 9). There is a substantial sediment adhering to the inner surface, very porous and reaching up to 1 mm in thickness, which consists primarily of silica (Table 11). This matter shows a layered microstructure typical of heavily weathered glasses (cf. Freestone 2001: 618-619), therefore its chemical composition is unlikely to offer any reliable estimate of the original content (fig. 99). However, the very presence of this sediment, improbable component of a freshly distilled liquid, may be taken as an indication that the instrument in question is a cucurbit rather than a receiver.

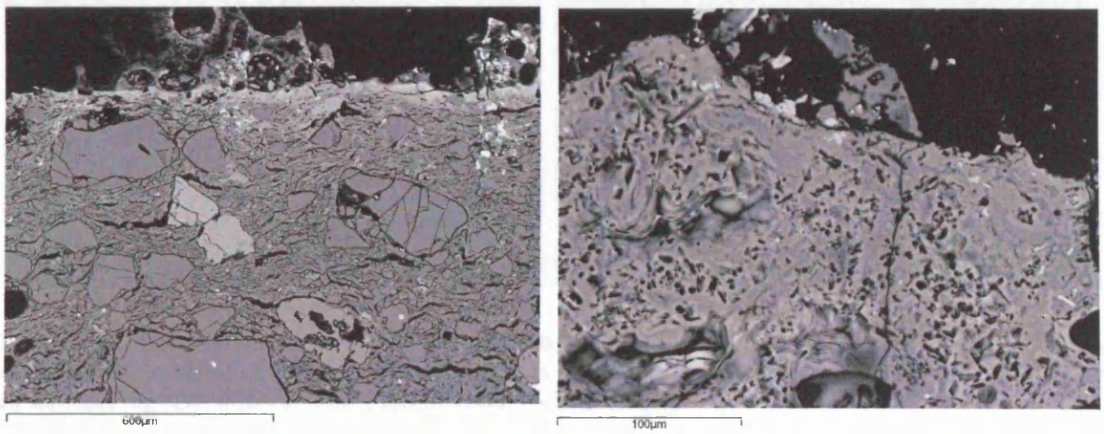


Figure 99. Left, ceramic fabric of the cucurbit, with shattered quartz grains and other silicate inclusions, and a residue adhering to the inner surface (top). Right, detail of this residue, with a sublayered structure typical of weathered glasses (OB n002/s1, BSE, left at 100x, right at 800x).

The last distillation apparatus analysed is a funnel-shaped instrument with a long neck (OB n005), of which approximately ten were found, interpreted by Soukup and Mayer (1997: 68-69) as a 'trumpet', a piece of equipment used for distillation by descension. Its fabric is also comparable to that of the cucurbit and the capital, albeit with an even lower alumina content and a higher occurrence of small zircon inclusions (Table 9). The inner surface shows evidence of repeated smoking, probably secondary, and some adhering residue which in some instruments appears clearly slipping down the slope of the funnel. In the analysed specimen, this sediment appears separated in two layers, the one at the surface being richer in iron oxide, and the one beneath richer in silica (figs. 100-101; Table 11). Nonetheless, this segregation may be the result of post-depositional weathering, as the structure in subparallel bands, characteristic of the hydrated silica layer in corroded glasses, is also noticeable here.

Figure 100. Two of the 'trumpets' from Oberstockstall. Scalebar is 15 cm.

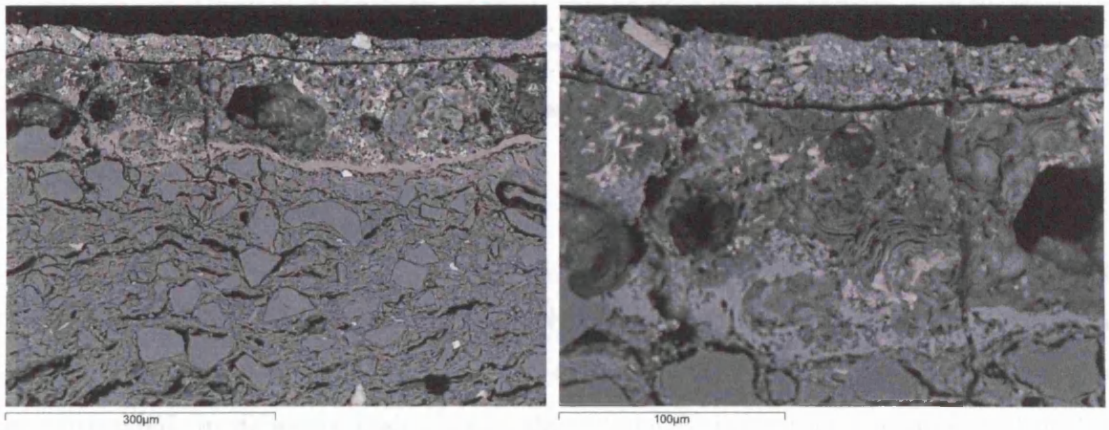


Figure 101. Left, fabric of a 'trumpet' with adhering residue on the inner surface (top). Right, detail of this residue showing two distinct layers and subparallel bands, separated probably due to weathering (OB n005/s1, BSE, left at 200x, right at 500x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO
OB n004 ('sandbath')	0.2	0.7	32.0	56.4	0.1	-	-	1.8	0.9	1.4	6.4
OB 17 (capital)	0.3	0.7	26.0	65.6	0.2	-	-	1.7	1.1	1.4	2.9
OB n002 (cucurbit)	0.5	0.4	23.1	66.7	0.2	-	-	5.2	0.4	1.1	2.4
OB n005 ('trumpet')	0.3	0.9	20.9	69.6	0.1	-	-	2.0	1.6	1.6	2.9
Crucibles	0.2	0.7	32.4	57.3	0.3	-	-	2.1	0.7	1.3	5.0
Scorifiers fabric A	0.8	0.5	31.9	55.3	0.3	≤0.5	≤0.1	3.3	0.6	1.3	5.4
Scorifiers fabric B	0.2	0.5	31.8	59.8	0.2	-	≤0.2	1.9	0.6	0.6	4.3

Table 9. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of distillation and sublimation instruments from Oberstockstall. The bottom rows present the average compositions of crucibles and scorifiers for comparative purposes.

	Al_2O_3	SiO_2	K_2O	CaO	FeO	CuO	PbO
OB 17 (capital)	1.2	34.4	-	0.7	-	4.5	59.6
OB n004 ('sandbath')	4.5	27.9	0.7	1.0	2.8	-	63.1
OB n003 (orange pot)	2.3	33.2	0.2	1.7	4.9	-	57.8

Table 10. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the glazes covering the inner surfaces of some of the instruments.

	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	MnO	FeO	As_2O_3	PbO
OB n002 (cucurbit)	-	0.5	4.1	88.0	2.0	2.4	-	1.8	0.6	0.7
OB n005 inner ('trumpet')	1.8	1.5	5.3	79.3	3.9	2.9	1.4	3.1	-	0.8
OB n005 outer ('trumpet')	-	0.4	5.7	32.8	0.8	-	0.6	60.2	-	-

Table 11. Average bulk chemical composition by SEM-EDS, normalised to 100 wt%, of the residues left inside the cucurbit and the 'trumpet'.

b) Ceramic containers

The sherd OB n003 (F.N. 38) is a fragment of the body of a thin (~4 mm), orange, internally glazed pot with a relatively big diameter and noticeable grooves from the potter's wheel, although the exact shape of the original artefact cannot be reconstructed. Its fabric is analogous to that of the non-graphitic scorifiers (fabric B), containing about 40 vol% of small, subangular, moderately spheroid silicate inclusions – mostly consisting of quartz, some feldspar, amphibole, iron oxide and mica –, together with some argillaceous inclusions that may be interpreted as grog, and minute ilmenite, rutile and zircon. It shows very little vitrification. The composition of the ceramic matrix is nearly identical to that of the scorifiers, only showing a slightly higher lime

content (Table 12). The glaze is a lead silicate layer, slightly thicker (~200 μm) and richer in silica and iron oxide than the one on the 'sandbath' instrument (fig. 102; Table 10).

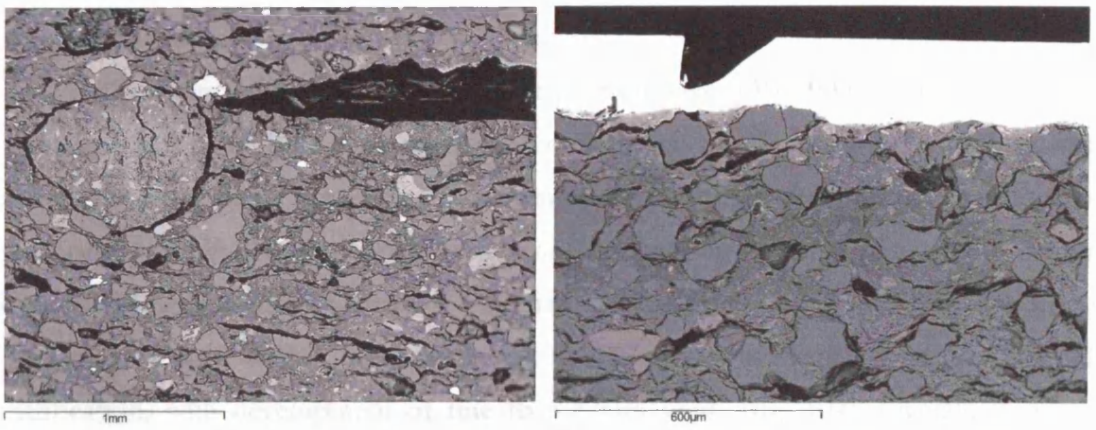


Figure 102. Two views of the fabric of a ceramic container. In the left image, the silicate inclusions may be noticed, as well as a big grog inclusion next to a large crack. In the right one, the interface between the ceramic and the inner glaze is shown. Note the limited vitrification (OB n003/s1, BSE, left at 50x, right at 100x).

Another comparative sample was taken from one of the several unstamped black jars, namely a 155 mm high cylindrical pot with slightly wide shoulders, a convex rim (162 mm in diameter) and a vertical handle (OB 389). The core of the fabric is light grey, while the surfaces appear burnished and smoked, hence showing a characteristic black metallic lustre, comparable to that described for the unused crucibles (fig. 103). Interestingly, identical pots were found in the estate of the apothecary in Krems, some 20 km from Oberstockstall. Historical records have revealed that Dr. Wolfgang Kappler, who owned the apothecary since 1527, maintained a close personal and professional relationship with the

Figure 103. Black unstamped pot OB 389. Scalebar is 15 cm.

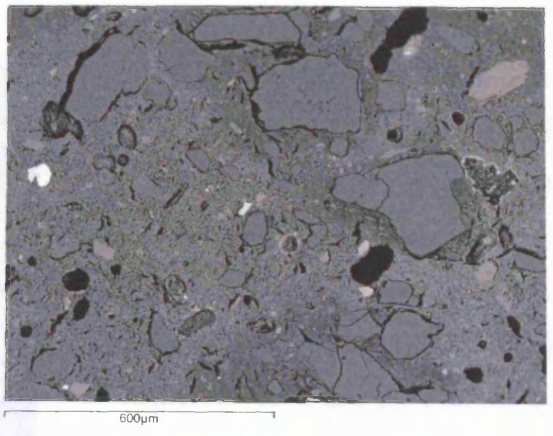


Figure 104. Fabric of the unstamped black pot, similar to that of the glazed one (see fig. 102), but showing more vitrification (OB 389/s1, BSE, 100x).

Trenbachs working in Oberstockstall. When Christoph von Trenbach died in 1549, he owed 4000 fl. to Dr. Kappler, which had to be disbursed by Urban, Christoph's brother. These facts had led to hypothesise that these pots could have been used as containers for the trade of chemicals (von Osten 1998: 55).

The fabric of this pot is akin to the previous one (OB n003), although no grog inclusions were identified. The nature and distribution of the mineral inclusions is the same, although these are slightly less abundant, and the chemical composition is analogous (Table 12; the main differences appearing in the lime and sulphur concentrations, relatable to burial contamination). Only the surface finish and the firing conditions clearly differ. In addition, this paste shows a more advanced stage of vitrification, with development of fine to medium voids (fig. 104). Overall, it seems reasonable to assume a common origin.

Figure 105. Left, dark ware. Right, detail of the Obernzell stamp on the rim (OB 362).

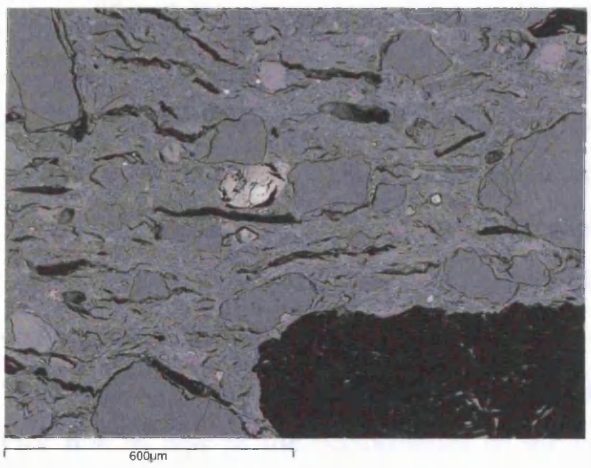


Figure 106. Fabric of a stamped pot, showing silicate inclusions together with some residual graphite (bottom right) (OB 389/s1, BSE, 100x).

The other dark ware amongst the comparative samples is a sherd (OB 362) from a big oblong pot, 240 mm high, with a convex rim bearing one of the typical stamps from Obernzell. The surface of this vessel is neither as dark nor as smooth as that of the previous one, although it also shows a smoked finish (fig. 105). In the core, the colour of the paste is

buff, therefore one may presume an oxidising atmosphere in the kiln, with the more reducing smoking taking place towards the end of the firing. The vitrification is very low, and the orientation of the clay minerals is still noticeable. The fabric contains numerous subspherical silicate inclusions (30-40 vol%), mostly quartz fragments, the biggest of which (~0.5 mm) appear shattered. Also iron oxide, feldspar, amphibole, ilmenite and rutile were identified as mineral inclusions, together with mica, which seems to occur more frequently than in the previous samples. Interestingly, also some (~5 vol%) scattered graphite fragments are present, sometimes intergrown with silicates (fig. 106). Due to their low concentration, and bearing in mind that naturally-bearing graphite clay banks were available in early modern Obernzell (Bauer 1983> 29), this graphite is deemed a residual inclusion rather than artificial temper. Compositionally, the ceramic matrix appears poorer in alumina and richer in iron oxide than that of the other black pot (Table 12).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO
OB n003 (orange)	0.2	0.5	31.8	59.5	0.2	-	-	1.2	1.8	0.7	4.2
OB 389 (unstamped)	0.3	0.9	31.8	55.0	0.2	1.3	-	1.7	3.7	0.7	4.4
OB 362 (stamped)	0.2	1.0	27.1	59.0	0.2	-	-	1.7	1.0	1.4	8.3
Crucibles	0.2	0.7	32.4	57.3	0.3	-	-	2.1	0.7	1.3	5.0
Scorifiers fabric A	0.8	0.5	31.9	55.3	0.3	≤0.5	≤0.1	3.3	0.6	1.3	5.4
Scorifiers fabric B	0.2	0.5	31.8	59.8	0.2	-	≤0.2	1.9	0.6	0.6	4.3

Table 12. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of some big vessels from the Oberstockstall assemblage. The bottom rows present the average compositions of crucibles and scorifiers for comparative purposes.

c) Lute

The last ceramic type from the Oberstockstall assemblage presented as comparative material is the lute, used for coating cucurbits and receivers – thereby minimising the heat shock and preventing the loss of the contents in the event of cracking –, sealing the joints between matching pieces of equipment, or shutting the mouths of crucibles or other vessels for reactions in closed atmospheres. As noted by Biringuccio, the lute had to be made "from a clay that is not viscous by nature, does not crack through shrinkage on drying, and is fire-resistant" (Smith and Gnudi 1990: 197). Other desirable properties were malleability and resistance to attack by acids. It had a vital role to play in the performance of the equipment and the efficient execution of several operations. Significantly, chymists often referred to it as *lutum sapientiae*, which

would translate as 'solder of wisdom', and contemporary treatises devote several pages to detail different recipes (cf. Thomas 2001).

Both the lute layers adhering to the outer surface of crucible OB 495 and cucurbit OB n002 contain a large (~50 vol%) proportion of mineral inclusions, but all of them are very fine, suggesting that the clay was sieved, as was common practice at the time. These consist mostly of quartz and potassium feldspar, as well as other silicates and mica (fig. 107). There is relatively little visible binding glassy phase between the clay and minerals, and the relatively high porosity may have originated from an organic binder or charcoal dust which has burned out. Compositionally, the clay matrix appears in both cases poor in alumina and very rich in lime (Table 13). The latter might be due to the deliberate addition of unslaked lime, as recorded in one of the recipes provided by Ercker (Sisco and Smith 1951: 153), and said to be particularly resistant to chemical attack.

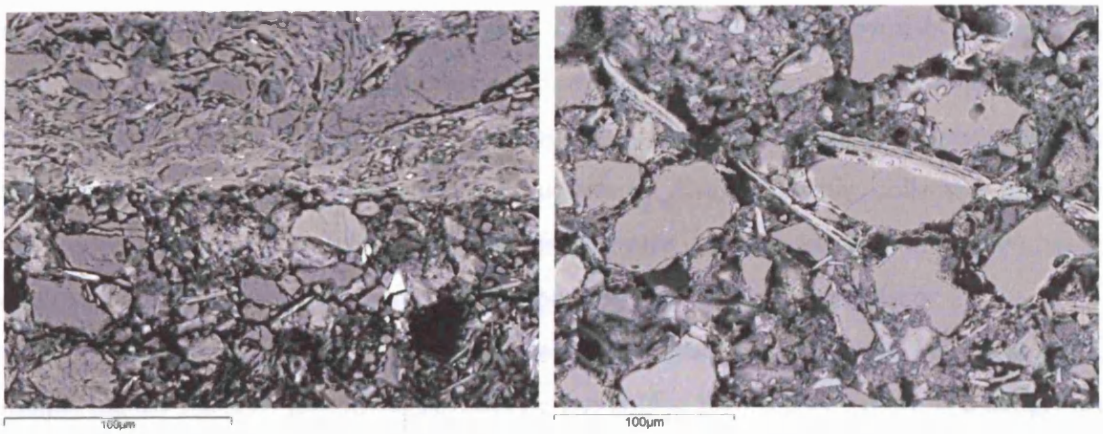


Figure 107. Left, interface between the body of a cucurbit (top) and the lute adhering to the outer surface. Right, detail of the lute attached to a triangular crucible (BSE; left, OB n002/s1 at 500x; right, OB 495/s2 at 400x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO
Lute crucible OB 495	0.5	4.6	11.8	44.0	0.9	0.6	0.8	0.8	24.3	1.0	10.8
Lute cucurbit OB n002	0.7	3.6	9.2	51.6	0.4	-	0.4	4.2	25.5	0.3	4.0

Table 13. Average chemical composition by SEM-EDS, normalised to 100wt%, of ceramic matrices of the lute samples from two different instruments.

6.5.2. Ceramic materials in the Oberstockstall laboratory: an overview

The detailed analyses of the crucibles, scorifiers and cupels from Oberstockstall, in conjunction with the examination of comparative material from the same assemblage, allow a more comprehensive approach to the technical standard and the choice of ceramics in the laboratory. Due to the lack of trace elemental compositions or exhaustive petrographic analyses of thin sections, the grouping and provenancing of some of these instruments has to remain tentative. Nonetheless, the available information serves as a basis for some general interpretations, which will be expanded in the next section after the comparison of these with ceramic materials from other sites.

In broad terms, two clearly different fabric groups are represented. These are, on the one hand, the high-magnesia, high-lime material used as lute and as a binder for the bone ash in the cupels; and, on the other hand, the high-alumina clays used for all the rest of the equipment (Table 14). It is worthwhile remembering that the lute and the cupels are, in all likelihood, the materials manufactured by the chymists themselves, whereas the degree of standardisation of the rest of the instruments clearly points to a more centralised and specialised production. Thus, the analytical data suggests that the artisans working in Oberstockstall used dolomitic clays, possibly collected in close vicinity, whilst other clay banks, probably from more distant sources (see below), were exploited by specialised potters in order to produce the rest of the artefacts.

Within the group of high-alumina ceramics, some subgroups can be demarcated, mainly on the basis of the different Si:Al:Fe ratios (fig. 108). Firstly, there is a subgroup of fabrics noticeably similar to those identified in crucibles and scorifiers. This includes the orange-glazed container OB n003 and the unstamped black jar OB 389, notably similar to some scorifiers (fabric B), together with the 'sandbath' instrument OB n004, which resembles particularly the paste of the crucibles. As discussed in the section on scorifiers (6.3.3), there are reasons to believe that the crucibles and scorifiers in Oberstockstall were supplied by the same producer, who used a range of similar clays, perhaps after refining them. By association, these other vessels would belong to the same group (Table 14).

The stamped black container OB 362, undoubtedly produced in Obernzell, is distinct in that is shows slightly lower alumina and higher iron oxide concentrations, as well as residual inclusions of graphite and more abundant mica. Furthermore, the paste is more buff than grey, and its surface finish is not so smooth, hence it is tempting to

suggest a different provenance. This idea will be developed in the next chapter, in the light of more analytical data.

Finally, another subgroup could be made with the ceramic fabrics of the sublimation capital (OB 17), the cucurbit (OB n002) and the 'trumpet' (OB n005). When compared to the ceramics ascribed to the Obernzell producer, these fabrics are characterised by the lower alumina and iron oxide levels, the corresponding higher silica content and, in the case of the capital, a distinct glaze (Table 14). Perhaps these three instruments, all of them specialised and directly related to wet chemical operations, were not amongst the range of types produced by the potters who made the high-temperature ceramics and other black wares, and the chymists working in Oberstockstall obtained them from a different manufacturer. Optically similar, unglazed and green-glazed buff fabrics, have been identified in ceramics excavated in Linz (Upper Austria) and held at the *Landesmuseum* (S. von Osten, pers. comm. 2004), and it is possibly in this region where we should be looking for the producer of distillation wares. The only apparatus from Oberstockstall related to distillation and not included in this category of fabrics is the 'sandbath' instrument. In the absence of any archaeological or historical evidence of such an unusual type, and considering its relatively easy manufacture, it could be suggested that this instrument type was made locally at the special request of the Oberstockstall chymist. It is worth noting that the glaze on this artefact is also different from those on the distillation instruments.

Manifestly, this hypothesis cannot be ascertained before much more historical and archaeometric research is carried out. In the meantime, one should also consider the possibility that all the comparative material analysed, including the distillation equipment, was produced by a single group of potters, who resorted to a range of clays much wider than believed here. Should this be the case, then it should be stressed how the high-temperature ceramics, namely crucibles and scorifiers, invariably fall within the alumina-rich end of the spectrum.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO	FeO
Cupel binder A	2.2	7.0	10.8	68.5	-	-	-	5.9	2.7	-	4.0
Cupel binder B	1.8	16.1	10.2	64.2	-	-	-	2.3	5.7	-	4.2
Cupel OB 937	-	4.5	7.8	28.0	-	8.8	0.9	2.6	31.1	-	10.9
'Bone ash' OB 925	-	4.7	3.1	18.1	-	0.4	-	1.7	67.2	-	2.4
Lute crucible OB 495	0.5	4.6	11.8	44.0	0.9	0.6	0.8	0.8	24.3	1.0	10.8
Lute cucurbit OB n002	0.7	3.6	9.2	51.6	0.4	-	0.4	4.2	25.5	0.3	4.0
OB n004 ('sandbath')	0.2	0.7	32.0	56.4	0.1	-	-	1.8	0.9	1.4	6.4
Crucibles	0.2	0.7	32.4	57.3	0.3	-	-	2.1	0.7	1.3	5.0
Scorifiers fabric A	0.8	0.5	31.9	55.3	0.3	≤0.5	≤0.1	3.3	0.6	1.3	5.4
Scorifiers fabric B	0.2	0.5	31.8	59.8	0.2	-	≤0.2	1.9	0.6	0.6	4.3
OB n003 (orange)	0.2	0.5	31.8	59.5	0.2	-	-	1.2	1.8	0.7	4.2
OB 389 (unstamped)	0.3	0.9	31.8	55.0	0.2	1.3	-	1.7	3.7	0.7	4.4
OB 362 (stamped)	0.2	1.0	27.1	59.0	0.2	-	-	1.7	1.0	1.4	8.3
OB 17 (capital)	0.3	0.7	26.0	65.6	0.2	-	-	1.7	1.1	1.4	2.9
OB n002 (cucurbit)	0.5	0.4	23.1	66.7	0.2	-	-	5.2	0.4	1.1	2.4
OB n005 ('trumpet')	0.3	0.9	20.9	69.6	0.1	-	-	2.0	1.6	1.6	2.9

Table 14. Comparative presentation of the average chemical composition of the ceramic matrices of different artefact types recovered in Oberstockstall and discussed in the text. Bold horizontal lines separate clearly different clays; the double grey horizontal line demarcates two slightly different clay matrices within the same broad fabric type.

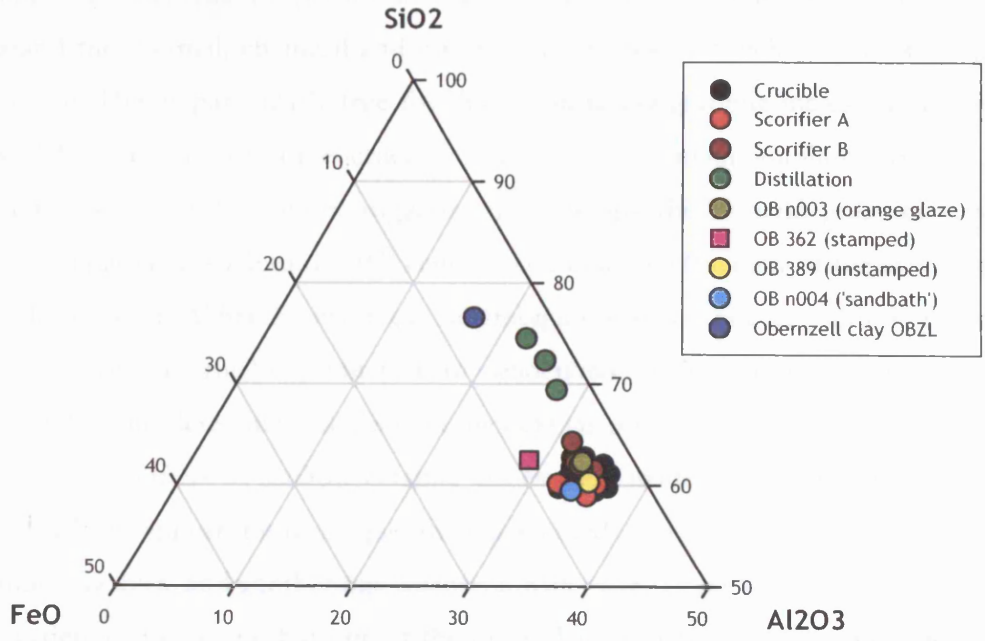


Figure 108. Ternary diagram plotting the SiO_2:Al_2O_3:FeO ratios of the crucibles, scorifiers and comparative ceramic material from Oberstockstall. Note the tight clustering of most artefacts, except for the clay sample from Obernzell, the distillation equipment and the stamped pot from Obernzell.

6.6. Summary

The assemblage from Oberstockstall comprises a very comprehensive array of state-of-the-art chymical equipment, as used in a 16th-century laboratory. Most of the utensils represented are known from contemporary written sources, and attest the conduction of both wet and dry chemical operations involving the assaying and/or refining of noble metals, but probably other processes as well.

For this thesis, the focus of the analytical work was placed on the technical ceramics used for high-temperature operations – namely crucibles, scorifiers and cupels –, although other ceramic objects from the same assemblage were also analysed, in order to allow comparisons and to provide a more comprehensive picture of the supply and use of different wares. The formal and material properties, and the performance characteristics, of these artefacts were discussed paying special attention to the choices made by the people who manufactured and used them. This allows a preliminary insight into the *chaîne opératoire* of this laboratory, illustrating not only how different tools related to each other in the provision and use of chymical equipment, but also how this laboratory engaged with the surrounding natural and cultural world.

The instrument most heavily represented in the assemblage is the triangular crucible. The material properties of these vessels would generally be adequate to withstand the thermal, chemical and mechanical stresses to which they were subjected during use. This is particularly true for those containing graphite inclusions within the fabric. The presence of some crucibles that do not contain graphite, coupled with relevant historical information, suggests that perhaps the mineral graphite was not clearly recognised as such in the 16th century, and that the efficiency of the crucibles was not related to it. What seems more unambiguous is a concern with the external appearance of the crucibles, which were deliberately made smooth and black. These ideas will be considered in more detail in the next chapters.

The scorifiers or shallow ceramic dishes were made with different fabric types, two of which appear more frequently represented: one is identical to that of the graphitic crucibles, and another one tempered with grog. These were versatile tools used for a variety of processes, but one of the most characteristic ones was the oxidation of lead bullion in order to concentrate the noble metals possibly contained within. Such reactions would convey a substantial challenge to the stability of the dishes, owing to the aggressiveness of hot lead oxide to the siliceous ceramic. The use of scorifiers would

have allowed saving the more sophisticated bone ash-based cupels, but it required swiftness and dexterity in interrupting the reaction before the chemical attack into the fabric became catastrophic. In fact, several scorifiers in the assemblage seem to have failed due to the massive penetration of lead oxide.

The analyses of the cupels from Oberstockstall tender several interesting findings. In contrast to the rest of the ceramic equipment, these vessels were probably made at the laboratory. Furthermore, their manufacture entailed two steps not recorded in contemporary written sources: on the one hand, the mixing of the bone ash with a sizeable part of clay and/or another lime-rich material, which detrimentally affected the absorption capability of the cupels; on the other hand, the recycling of the cleaner parts of used cupels for the production of new ones.

From a wider perspective, and based on the chemistry and mineralogy of the pastes, it can be hypothesised that the chymists working in Oberstockstall obtained equipment from a variety of producers. Stamped black pots were acquired from Obernzell potters; other black wares, ordinary glazed pottery, crucibles and scorifiers seem to come from a different source, possibly within Austria (see below, section 7.5); finally, distillation equipment, with the exception of the unusual 'sandbath instrument', forms a distinct group and may originate from yet a different source.

Only forthcoming work focussing on residues of use, and perhaps expanding the range of artefact types considered, will allow a detailed characterisation of the reactions conducted in the Oberstockstall laboratory. At present, however, and notwithstanding the fact that many operations would certainly fall outside this scheme, a generic model can be suggested for the sequence of operations involving high temperatures: at the outset, mineral samples were processed in reducing conditions within the black triangular crucibles, possibly with the addition of lead; subsequently, the lead-dominated bullion was oxidised in a scorifier, in order to reduce its size and concentrate the noble metals; finally, the smaller button was refined in a white cupel.

In 1477, when addressing the production of the philosopher's stone, Thomas Norton discussed at length the importance of colour. One of his observations may be of relevance here, as similar ideas might have been at stake in Oberstockstall when conducting reactions that started in *necessarily* black crucibles and ended in *necessarily* white cupels:

"But White and Black, as all men maie see,
Be Colours contrary in most extremitie:
Wherefore your warke with Black must beginn,
If the ende shulde be with Whitenes to winn." (Norton [1477] 1652: 56)

7. The black crucibles from Bavaria

"Diamonds shine more brightly foiled by black velvet."
Sai Baba (1838-1918)

7.1. Introduction

The investigation of the wider context of production and use of the triangular crucibles found in Oberstockstall led this research to the large graphite deposits centred in Bavaria and scattered through Bohemia and Upper Austria. With a few noteworthy exceptions (see below), remarkably little attention has been paid to the production of graphitic crucibles in this region, which took place in a large scale at least since the late Middle Ages (fig. 109). As will be discussed in chapter 8, this lack of research may have led to overplay the historical importance of the better-known Hessian crucibles, produced in Central Germany.

This chapter will start by reviewing the scarce available information regarding the production of black wares in general, and crucibles in particular, placing special emphasis on the village of Obernzell, which seems to have been the key nucleus of this industry. After that, the information will be completed with the comparative characterisation of crucibles and other ceramics produced in Obernzell and found at different sites. The focus will subsequently be placed on the interesting stamp exhibited by some of these wares. This chapter shall also address the provenance of the crucibles found in Oberstockstall, in the light of historical, archaeological and archaeometric data, before giving a final summary with perspectives for future work.

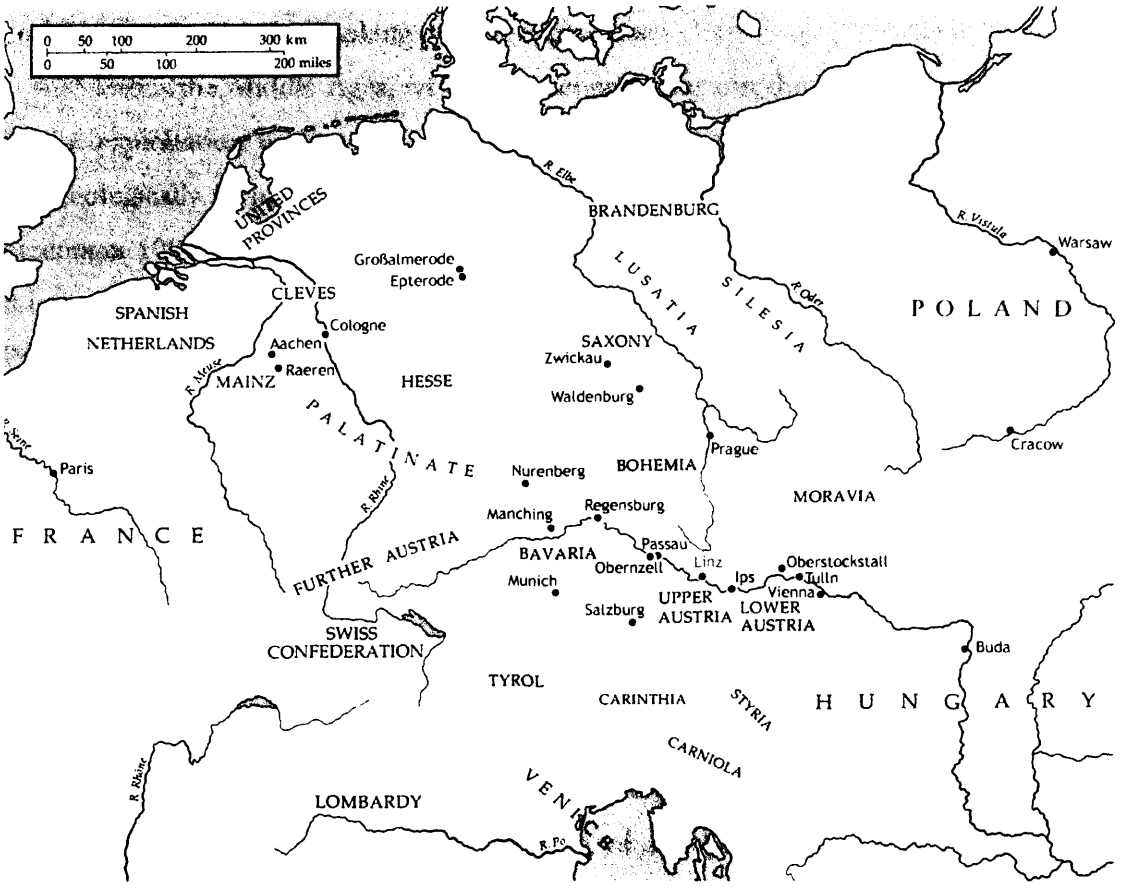


Figure 109. Map of the Holy Roman Empire in the 16th century, with indication of the main places discussed in the text (after Roberts 1991: 149, modified).

7.2. Crucible production in Bavaria: historical information[25]

The production of graphitic and other black wares in Bavaria dates back to Prehistory, as attested by the famous graphitic vessels recovered in the Celtic oppidum of Manching (cf. Kappel 1969; Tylecote 1982). Although it seems clear that graphitic ceramics were valued in some way – clays or finished pottery were obtained as far as 200 km from Manching –, the motivations behind this choice probably rested outside our modern technical appreciation of the material properties of graphite, as suggested by the

[25] I am enormously grateful to Ingolf Bauer (Director of the *Bayerisches Nationalmuseum*) and Rudolf Hammel (ex-mayor of Obernzell), who very keenly shared with me their precious research on the history of crucible production in Obernzell and the surrounding region. They gave me the lead to most of the bibliography cited in this section, including several unpublished documents which they transcribed, and patiently answered my numerous questions. Although it is hoped that this thesis will contribute to redress the traditional disregard of the importance of this industry, I must credit these researchers as the true forerunners, and somewhat apologise for using their raw data for interpretations that differ from theirs. An array of documents relevant to this history is to be found in the *Bayerisches Hauptstaatsarchiv München*, the *Marktarchiv Obernzell*, and the *Staatsarchiv für Niederbayern in Landshut*.

apparently asystematic use of graphitic and non-graphitic clays for metallurgical crucibles as much as for cooking pots or containers.

From the Middle Ages, written documents bear out the keen interest of potters in the exploitation and use of graphitic clays in the region, as is also documented archaeologically (Schultheiß 1956; Holl 1976; Bauer 1976; Pittioni 1976; Pittioni 1977; Steininger 1980; Endres 1982; Bauer 1983; Kainz and Ratsuny 2000; Hammel 2002). At this point, a small village plays a central role. Obernzell, belonging to the archdiocese of Passau, is located in Upper Bavaria (SE Germany), on the right bank of the Danube River and near the border with present-day Austria. It lies in the heart of the largest deposits of graphite in Europe, and this mineral wealth is still exploited today.

Archaeological remains document a wide variety of black wares manufactured in Obernzell, mostly oblong pots of different sizes, with or without handles, and shallower pans. Additionally, some glazed, oxidised fired, earthenwares were produced. The most common types were traded along the Danube and were widely sold in Austria although, interestingly, they found competitors producing similar black pottery. In 1509, trade regulations in nearby Regensburg banned the commerce of any pottery produced outside this district, with the only exception of the black ceramics from Obernzell (Bauer 1983: 29). This indicates that the Obernzell wares were established and recognised as special articles, perhaps not easy to produce or obtain anywhere else. Only a few years later, in 1516, archbishop Wiguläus of Passau establishes a bylaw, perhaps normalising an earlier custom, whereby only potters belonging to the Obernzell guild were allowed to exploit and sell '*Eisentachen*' or 'graphitic' clay (see below). This ordinance would be confirmed by Leopold I in 1613 (Bayerisches Hauptstaatsarchiv München Passauer Urkunden 1869, as cited in Bauer 1983: 29).

It is not clear when the large-scale production of crucibles started. In 1549, written documents prove that Obernzeller crucibles were used in the Linz Mint (Austria) (Marktarchiv Obernzell, transcr. R. Hammel 2002). By the early 17th century – although this may have started earlier –, we see them delivered to the Royal Mints in Vienna, Munich and Prague (Bauer 1983: 30). At this time, the crucible industry appears as a solid and prosperous trade, with increasing numbers of apprentices in the many workshops scattered across the region.

Even though Obernzell potters had the prerogative of exploiting the local graphitic clays, smaller occurrences may have been exploited elsewhere, and there is evidence of clays being both exported from, and imported into, Obernzell (Kainz and

Ratsuny 2000; Hammel 2002). This creates a very confusing panorama for archaeologists trying to provenance the crucibles and other wares, in the lack of ceramic waste from production centres that could serve as unequivocal reference material. Whatever the case, it is patent that crucibles were produced primarily in Obernzell, but also in other villages scattered within Bohemia, Bavaria and Upper Austria. For example, a 1548 document mentions crucible makers working in Heroldsberg, Nuremberg (Schultheiß 1956: 27-28). Black crucibles were also produced in Ips, another town by the Danube, closer to Vienna. These have become historically more famous than those from Obernzell, despite the quite remarkable fact that, at least in the 18th century, Ipsian crucible makers had to import clay from Passau and Bavaria (Gaspari 1797: 591). In addition, "Vienna crucibles" are known to several 16th-century writers (Sisco and Smith 1949: 110; Smith and Gnudi 1990: 72), although these may have been simply traded from there. Overall, a range of crucible makers seem to have worked in the region and, as far as it is known, all of their products share one attribute: their black colour (Beckmann 1780, as cited in Stephan 1995: 31; Beckmann 1787, as cited in Bauer 1976: 14-15)[26].

Many of these crucibles bear makers' stamps on the base, although it is not always easy to attribute these to a certain producer. Given the special importance of Obernzell as a producer of crucibles and other black wares, it seems unlikely that this village imported similar pottery types manufactured elsewhere. Accordingly, some stamps frequently identified in pottery found in Obernzell are assumed to have originated there (see sections 6.1 and 6.5). Other stamps, such as the 'T' seen in the crucibles from Oberstockstall, have not been recorded in Obernzell, and therefore their ascription remains more difficult (see section 7.5).

Other uncertain questions are the reason why graphitic pottery became so highly appreciated, or when potters started to artificially add crushed graphite to the clay mix, rather than resorting to naturally graphitic clays. Traditionally, it has been assumed that graphitic clays were chosen for reasons akin to the present-day ones, such as their thermal refractoriness, conductivity, or impermeability (e.g. Kappel 1969; Duma and Ravasz 1976; Bauer 1983). Nevertheless, there is no clear evidence to support this claim as far as early modern times are concerned. As advanced in section 6.2.2, early documents repeatedly demonstrate the use of '*Eisentachen*' ('iron clay'), which in all

[26] Within the constraints of this project, it is not possible to investigate in detail all the producers of black crucibles and their interactions. This simply constitutes an attempt to set the grounds for future enquiry.

likelihood represents the dark, shiny, iron-like, naturally graphitic clays (Schultheiß 1956; Bauer 1976; Pittioni 1977; Bauer 1983). A slightly clearer reference is made by Libavius, when he mentions crucibles made of "*aschehaltigem Ipsener Ton*" or 'ash-rich clay from Ips' (Rex 1964: 12)[27]. In stark contrast, no reference is made to the much more abundant graphite deposits. Only from the late 18th century do we find explicit allusions to the mixture of clay and graphite during the paste preparation for crucible manufacture. In these sources, the term used for the temper is "*Wasserbley*" ('liquid lead', or 'lead water') (Beckmann 1780, as cited in Stephan 1995: 31; Beckmann 1787, as cited in Bauer 1976: 15). Again, the expression highlights the greasy and metallic appearance of the mineral, while it also suggests that, at least in its origins, some confusion may have existed as to its identification or categorisation. It is worth remembering that, during the Renaissance, the prevalent view of nature was that the prime matter could be modified by altering its dryness and heat, either naturally or artificially, and this led to ambiguous classifications of the natural world including earths, juices, gems, stones, rocks, metals, compounds and even the so-called 'semiminerals', which comprised quicksilver, sulphur, marcasite, alum or calamine. This was a matter of debate still in the 16th century, acknowledged by Biringuccio when he wrote that "I do not intend to [...] enter now this maze of speculation [and,] leaving aside opinions and disputes, I shall take only those of the said semiminerals that I know are useful" (Smith and Gnudi 1990: 77).

As noted before, most Renaissance and earlier treatise writers describe in detail the manufacture of crucibles, being aware that, as observed by Agricola, "not only potters, but also the assayers themselves, make scorifiers and triangular crucibles" (Hoover and Hoover 1950: 230). Their annotations include references to what they consider the most suitable clay banks, the temper, and even the firing (see discussion in chapter 10), but no explicit mention of graphite or graphitic clays is to be found in any of these sources. Overall, for the early modern period, it seems that some potters were particularly interested in the use of 'iron clay' for the production of crucibles and other black wares, but their products were only perceived and known by the customers as 'black pottery.' At some point, the link between this 'iron clay' and the 'liquid lead' must have been realised. In the context of increasing competition, some potters might have discovered this link but kept it secret, so that the full recognition of the triad

[27] Interestingly, Libavius cites Agricola's *De natura fossilium* as his source for this reference, but the adjective originally used by Agricola (Fraustadt and Prescher 1958: 52) was simply "*aschgrau*", *i.e.* 'ash-grey".

'graphite/black wares/good performance' may not have been widespread for some time. Before this, perhaps some potters and consumers were happy enough with black, lustrous pots, regardless of the exact nature of this 'blackness'.

7.3. The production and trade of black crucibles: archaeological and archaeometric information

Having briefly outlined the history of crucible production in Obernzell and the surrounding region, the available information may be completed with the archaeometric characterisation of the formal and material properties of some of these productions. Firstly, some samples of black wares recovered in Obernzell will be addressed comparatively. Secondly, analytical results will be presented from a graphitic crucible recovered amongst the laboratory equipment from Oxford. The subsequent discussion will also address some other black crucibles found in Britain and as far as America. It may be emphasised from the beginning that the provenance of all of these vessels, except for those recovered in Obernzell itself, had not been identified until the present work, which contends the ascription of most of them to the same Bavarian village. This stresses both the span of the trade of black crucibles in the post-medieval world, and the need of fundamental research in this field.

7.3.1. Black wares from Obernzell

Even though the four ceramic samples from Obernzell show a black, smoked surface, that of the crucible is particularly lustrous, as a result of a very smooth surface finish and a high graphite content. This vessel was recovered during building works in Obernzell, and tentatively dated to the 16th or 17th century (R. Hammel, pers. comm. 2003). The sample is the bottom half of a beaker-shaped crucible, about 70 mm diameter at the base, which bears no stamp. The wall is 12 mm thick. An orangish sediment covers part of the

Figure 110. Crucible sample from Obernzell (OBZL 01).

surfaces, including the fractured edges, thus denoting some post-depositional alteration (fig. 110). The ceramic matrix is light grey, but the general appearance of the paste is darker, due to the abundance of graphite inclusions. Approximately 50 vol% of the fabric is composed of graphite flakes, some of them rather big (up to 1 mm long), oriented parallel to the surfaces. In addition, there is 10-15 vol% of other mineral inclusions, subangular and up to 1 mm large (figs. 111-112). These are mostly quartz grains, but also potassium feldspar, iron oxides, apatite, amphibole, mica and rutile particles were identified. The ceramic shows an intermediate degree of vitrification, more advanced towards the surfaces. Part of the graphite inclusions on the outer surface have burned away, although it is not possibly to determine whether this is due to oxidation during original firing or use. Generally, the crucible 'feels' different from those from Oberstockstall in that it is thicker, heavier, and clearly richer in graphite, apart from not being triangular. The chemical composition shows a relatively high alumina content, together with a high iron oxide concentration (Table 15, p. 197).

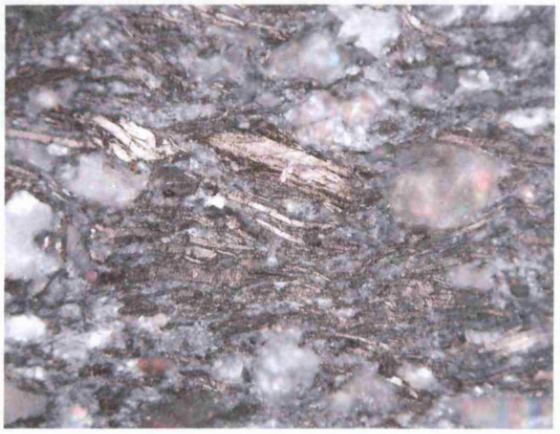


Figure 111. Detail of the fabric of the crucible from Obernzell, showing abundant graphite speckles and silicate inclusions (OBZL 01/s1, XPL, 50x, long axis represents ~2 mm).

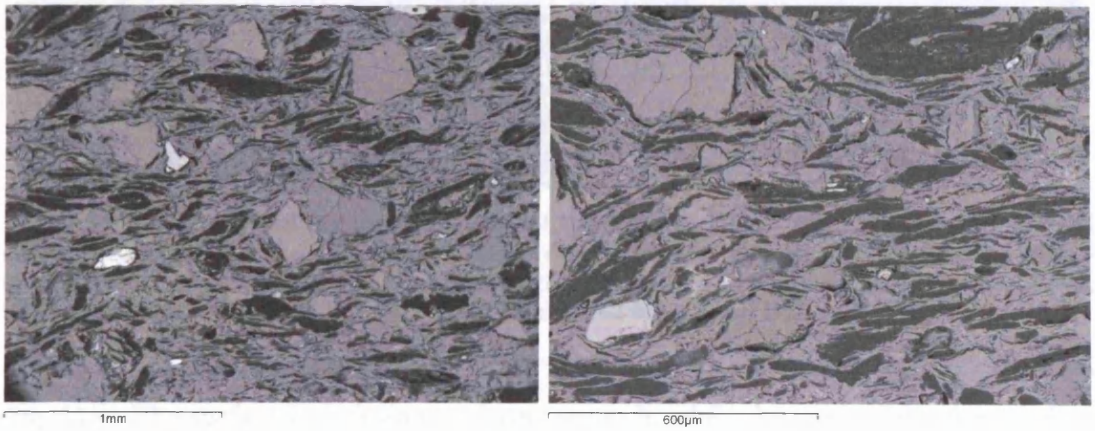


Figure 112. BSE photomicrographs of the ceramic fabric of the crucible from Obernzell. Note the remarkably high concentration of graphite inclusions (dark elongate phases), in addition to the silicate grains (OBZL 01/s1; left at 50x; right at 100x). Compare to figs. 41 (p. 114) and 47 (p. 116).

Figure 113. Lid fragment from Obernzell (OBZL 03).

The other ceramic materials from Obernzell include a vertical handle, a lid, and a rim fragment. The lid (OBZL 03) is 6 mm thick. It has a domed shape, although the original diameter cannot be reconstructed, and bears a small knob on the top (fig. 113). The paste is grey to buff in colour. It contains relatively abundant coarse inclusions, some larger than 1 mm. Apart from the more recurrent quartz, also feldspar, apatite, iron oxide, ilmenite and some mica are present, together with fragments of amphibole, the latter occurring more frequently than in any of the Oberstockstall artefacts. Under high magnification, the fabric is characterised by a very low vitrification and the profusion of very small (<100 μm) quartz inclusions (fig. 114). The elemental composition of the matrix is similar to that of the crucible, albeit with an even higher iron oxide concentration (Table 15, p. 197).

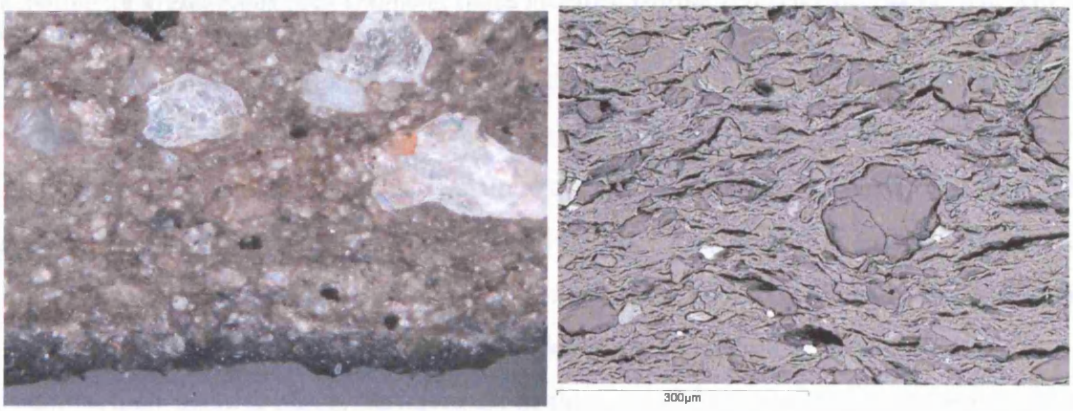


Figure 114. Two views of the ceramic fabric of the lid from Obernzell. Left, note the smoked surface (bottom) and the large size of some of the silicate inclusions. Right, under higher magnification, note limited vitrification and the abundance of very small quartz inclusions (OBZL 03/s1; left, XPL at 50x, long axis represents ~2 mm; right, BSE at 200x).

OBZL 04 is the fragment of the rounded rim of a relatively large, globular pot (fig. 115). The surface is dark grey and very smooth, while the core of the paste is buff. The thickness of the body is 6 mm. There is an elongate stamp on the rim, but the motif is not very deeply imprinted and cannot be identified. The fabric contains 40 vol% of rather coarse mineral inclusions, including quartz, feldspar, amphibole, mica, iron oxide and ilmenite, together with some argillaceous rock fragments (fig. 116). It shows an

advanced stage of vitrification, which may be explained by its relatively low refractoriness in an oxidising fire, as a result of its high iron oxide content, of above 10 wt% (Table 15, p. 197).

Figure 115. Rim fragment from Obernzell (OBZL 04).

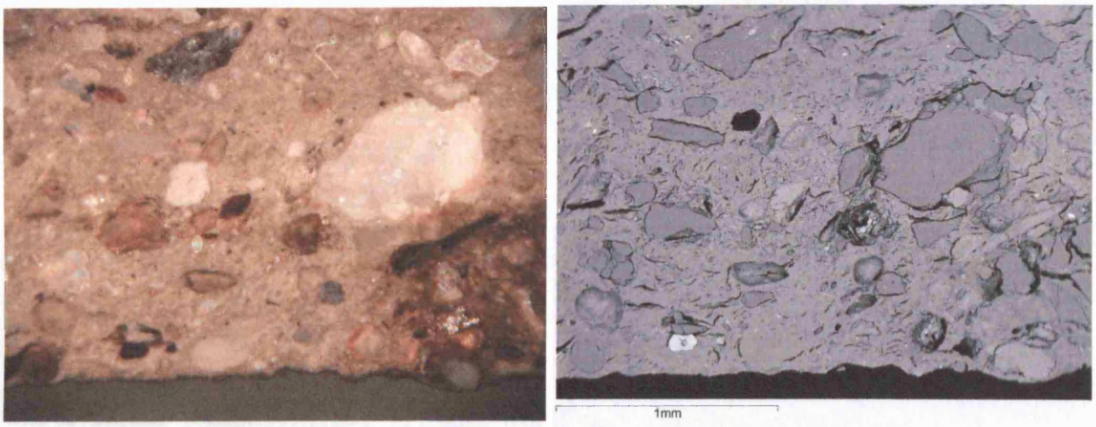


Figure 116. Two views of the same area of the ceramic fabric of the rim fragment from Obernzell. Note the very thin smoking (left image, bottom), and the rather coarse inclusions, including an argillaceous rock fragment (both pictures, bottom right) (OBZL 04/s1; left, XPL at 50x; right, BSE at 50x).

Finally, OBZL 02 is a vertical handle stamped with a cross – one of the typical symbols from Obernzell pottery, even though different from those recorded in the Oberstockstall assemblage (fig. 117). Mineral inclusions within the paste, which is light grey in colour, are very abundant (~50 vol%). They are, however, smaller than in the other domestic ceramics from Obernzell, as they rarely exceed 0.5 mm in diameter. The most noteworthy feature of this fabric is the abundance of dark grains of amphibole and biotite, which make it clearly different from the rest of the samples. In addition, quartz, feldspar, iron oxide and plagioclase were identified (fig. 118). The composition of the ceramic matrix is also different, especially in its phosphate and lime content, but these results are clearly distorted by abundant corrosion products that can be seen filling cracks and pores (Table 15).

Figure 117. Handle fragment from Obernzell (OBZL 02).

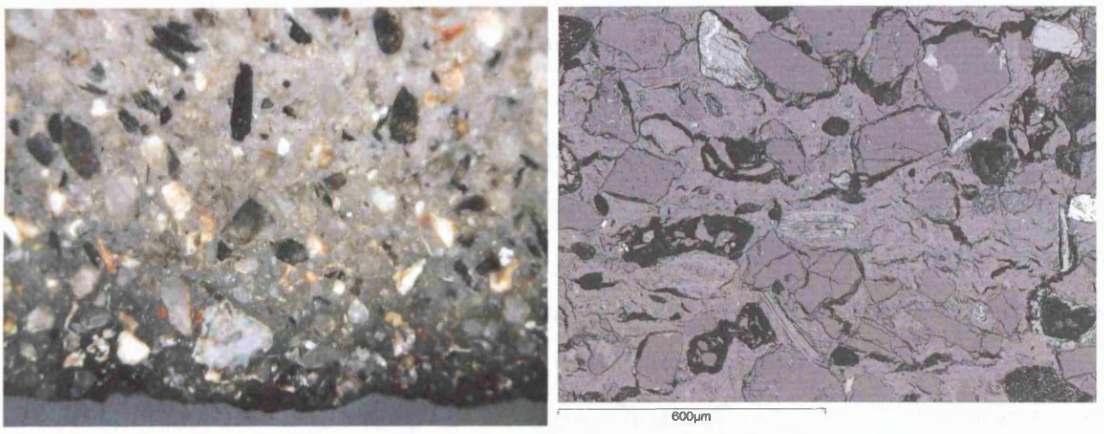


Figure 118. Two views of the ceramic fabric of the handle from Obernzell. Note the surface smoking (left picture, bottom), and the abundance of small inclusions, particularly mica and amphibole (dark in left image, light grey in right image). The deposition of corrosion products within porosities is also noticeable (OBZL 02/s1; left, XPL at 50x, long axis represents ~2 mm; right, BSE at 100x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO	FeO
OBZL 01 (crucible)	1.1	1.0	27.7	58.3	0.4	-	-	2.5	0.9	1.2	6.9
OBZL 03 (lid)	0.7	1.2	26.0	58.4	1.0	-	-	2.1	1.1	1.3	8.2
OBZL 04 (rim)	0.3	0.7	28.2	55.1	0.6	-	-	1.6	1.4	1.6	10.5
OBZL 02 (handle)	0.4	0.7	26.6	55.3	4.9	0.8	-	1.3	3.0	1.2	5.9
OX 1422 (crucible)	0.3	0.8	28.9	55.5	0.2	-	-	2.6	0.9	1.4	9.3
Obernzell clay	2.5	1.7	15.2	69.0	0.2	-	0.2	1.6	3.0	0.6	5.9

Table 15. Average chemical composition by SEM-EDS, normalised to 100wt%, of the ceramic matrices of the four analysed ceramics from Obernzell (OBZL), the graphitic crucible from the Old Ashmolean laboratory in Oxford (OX) and the clay sample from Obernzell.

Some general inferences may be drawn from the comparative characterisation of the ceramic sample from Obernzell. The most obvious one is the fact that, as in the case of Oberstockstall, only the crucible contains a significant amount of graphite inclusions. Again, this could be taken as an indication that the most suitable fabrics were reserved for high-temperature technical ceramics. This situation compels a reassessment of the existing black wares unquestionably produced in Obernzell and dated to the late medieval and early modern periods. In a visit to the *Keramikmuseum* in Obernzell, ordinary ceramics containing graphite seem the rule. However, the analytical approach suggests otherwise for the samples from both Obernzell and Oberstockstall. Given the similar external appearance of graphitic and non-graphitic black wares, and considering that graphitic pots may have been specifically chosen for exhibition in the museum, the presence of graphitic pottery in medieval and Renaissance material culture may have been overemphasised in historical and museum reconstructions. A global revision of this scenario would clarify our understanding of the selection of clays.

Only one sample of clay from Obernzell could be obtained for analysis, and it turned out to be a low-alumina clay, significantly less refractory (Table 15). While this illustrates that not all clays in the area were of the same quality, the use of alumina-rich clays for all the ceramics analysed suggests that the most refractory clays were not specifically reserved for artefacts to be used under high temperatures.

It is worth noting the specifics of the Obernzell ceramics when compared to those found in Oberstockstall. In terms of mineral inclusions, the fabrics from Obernzell are generally distinct in the higher abundance of biotite and amphibole inclusions, together with the virtual absence of zircon. Compositionally, the most notorious features are the slightly lower alumina and higher iron oxide concentrations. These characters are relevant for the discussion of the provenance of the crucibles from Oberstockstall, and will recaptured in section 7.5.

Finally, a special comment should be made on the high iron oxide level within the clay matrix of the crucible, insofar as it would affect its performance. As discussed before (section 6.2.4), even though the influence of this compound on the heat resistance of a vessel would depend on the redox conditions, this iron oxide content might have compromised the refractoriness of the clay matrix when used in an oxidising or moderately reducing furnace. Nevertheless, there is so much graphite in the paste that, even in the event of the clay matrix softening, the fabric would probably have

retained sufficient consistency. As to further material properties of graphite and other mineral inclusions, these have already been discussed (sections 6.2.2 and 6.2.4).

7.3.2. Black crucible from the Old Ashmolean Laboratory in Oxford

The heading of this section may appear paradoxal after a look at the graphitic crucible found in the Old Ashmolean laboratory, given that it shows a bright brown colour (fig. 119). Nonetheless, the current appearance of this vessel is clearly the result of its use in a very oxidising atmosphere, which caused the burning away of some surface graphite inclusions and the oxidation of the relatively abundant iron content of the clay matrix (Table 15, p. 197).

Figure 119. Graphitic crucible from the Old Ashmolean laboratory (OX 1422).

This crucible is unique within the assemblage, both in the stamp and the fabric characteristics. It is a wheel-thrown, beaker-shaped vessel, 100 mm high, with a wide mouth of 80 mm diameter. The body is 7 mm thick and the surface smooth. The thicker, flat, round base (43 mm in diameter) has a number 3 incised, together with a more complex stamp, about 20 mm long, consisting of a combination of a number four, a cross and two initials – LH or LK – inside an elongate cartouche. Although similar stamps have been recorded in other production centres, not only of pottery, this symbol in particular is exactly like others documented in Obernzell, even in the potter's or merchant's initials (fig. 120; see discussion in section 7.4).

The fabric of this crucible is characterised by an extraordinary abundance of graphite inclusions, above 70 vol%, many of which are very large (up to 1 mm long). Minor inclusions are potassium feldspar, quartz, mica, amphibole, rutile, zircon and iron oxide minerals. The clay matrix shows an advanced stage of extensive vitrification, with a high degree of interconnectivity and development of fine to medium voids. However, given that much of the fabric is composed of graphite, the vessel appears robust and must have been very stable in the fire (fig. 121). In spite of the brown surface colour, the oxidation is constrained to a very thin layer, and most of the graphite inclusions have remained unaltered (fig. 122).

The chemical composition of the clay matrix is very similar to that of the Obernzell crucible discussed above (Table 15, p. 197), and so are the mineral inclusions. These factors, in addition to the stamp, confirm the provenance of this crucible beyond question, and solve the original doubts regarding its ascription (Hull 2003; Bennett *et al.* 2000: 46).

Figure 120. Detail of the bottom of the crucible OX 1422, showing the typical Obernzell stamp and a number 3. Compare to figs. 124 and 125 (trace from Bennett *et al.* 2000, fig. 34).

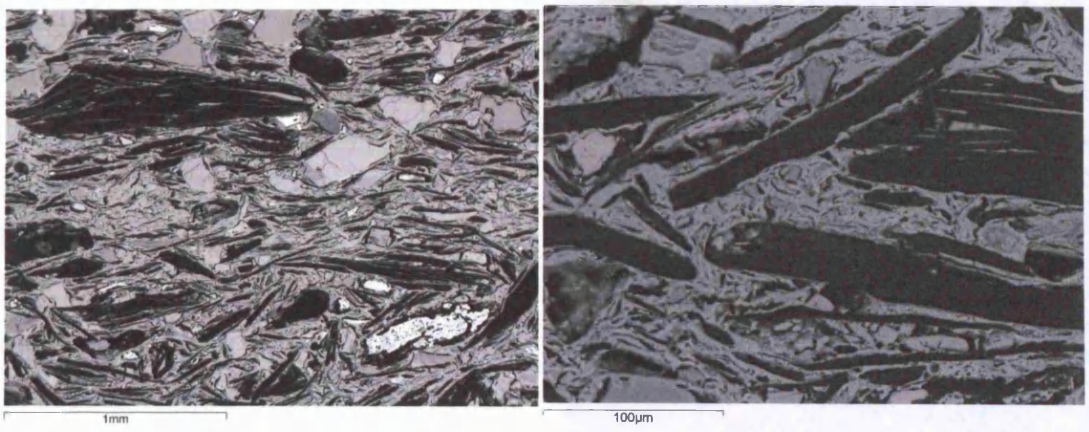


Figure 121. BSE photomicrographs of the fabric of the graphitic crucible from Oxford, showing the outstanding abundance of relatively large graphite inclusions, as well as some silicates. The high vitrification of the clay matrix is noticeable in the image on the right (OX 1422/s1; left at 50x; right at 400x).

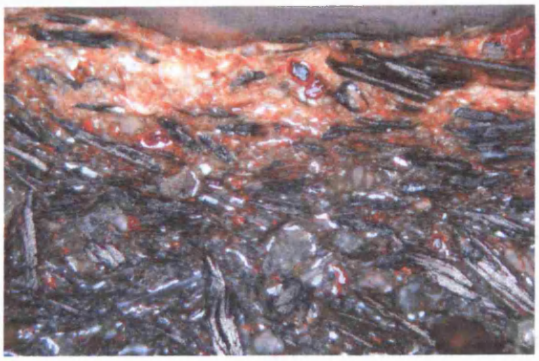


Figure 122. Cross-section showing the inner surface of the crucible, with an oxidised colour but limited burning of graphite inclusions (OX 1422/s1, sXPL, 100x, long axis represents ~1 mm).

7.3.3. Other examples of black crucibles

Oxford is not the only English location where dark graphitic crucibles were imported in the post-medieval period. In his catalogue of crucibles recorded in Britain, John Cotter illustrated several stamps "in the hope that they may eventually be identified" (Cotter 1992: 259). After the present study, some of the stamps found in these graphitic crucibles may be traced to Bavaria.

Firstly, two crucibles found in London, and nowadays kept in the Museum of London, may be addressed. Both of them seem beaker-shaped, although this cannot be confirmed as the top parts are missing. One of them (MoL 24859) was found near St. Paul's stairs. Only the bottom half of the vessel is preserved, which is very similar in appearance to the Obernzell crucible discussed above (OBZL 01, section 7.3.1), although bigger in size. It shows a very smooth black surface and a dark grey, graphite-rich, fabric. The base diameter is

Figure 123. Two stamped graphitic crucibles found in London (top, MoL 24859; bottom, MoL A730). Scalebar is 5 cm.

~80 mm, and the wall is 10 mm thick. The other one (MoL A730) was found near the Thames and has preserved about two thirds of its height (~145 mm), although it appears fairly weathered. Its base diameter is akin to the previous one, but the wall is slightly thinner (~8 mm) (fig. 123). The fabric is also very rich in graphite. Both of these crucibles are stamped on their bases with marks resembling that described above for the crucible from Oxford and identified as typical of the Obernzell producer, although with different initials (respectively, AH or AK, and KM) (fig. 124). They may thus be attributed to the same region although, perhaps, to a different maker and/or date. Unfortunately, none of the crucibles found in London could be dated.

Cotter also reports two graphitic crucibles recovered in Canterbury (Kent), both of them wheel-thrown and triangular. The first one has a height of 110 mm and a mouth width of 100 mm. It bears four finger-tip impressions at the base. The stamp in the base is akin to those just described, including an L (or inverted F) and a K (Royal Museum of Canterbury Acc. No. 4247, Brent Catalogue H129) (fig. 124). As in the previous case, no dates are available, but the combination of a graphitic paste and the characteristic stamp suggests Obernzell as the likely production centre.

The second crucible, found in Canterbury Barracks, is larger, with a height of 200 mm and a mouth width of 175 mm (Royal Museum of Canterbury Acc. No. 6795). Cotter (1992: 260) observed that a "brown oxidised coating covers the exterior", but this is likely an oxidation layer from use similar to that noted in the crucible from Oxford. The stamp in this vessel is also bigger (50 mm compared to the usual 25-30), and the symbol appears delineated more neatly, as if imprinted with a metallic, rather than a ceramic, die. The bottom part of the mark forms an anchor, and the initials are IK (fig. 124). Another stamp next to this shows a number 13, possibly a size grading. Undoubtedly, this crucible was made in the workshop of Josef Kaufmann and partners, a crucible making corporation in late 19th-century Obernzell (Bauer 1983: 32, and section 7.4).

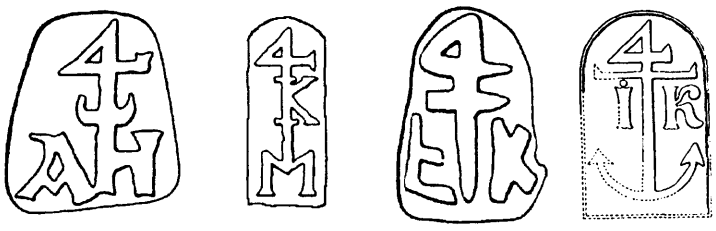


Figure 124. Stamps imprinted on the bases of graphitic crucibles. From left to right: MoL 24859, MoL A730, Canterbury 4247, and Canterbury 6795 (from Cotter 1992).

Crucibles with the same stamp of Josef Kaufmann, almost incontestably Obernzeller, have been found in Austria (Pittioni 1977). Another graphitic crucible has been recovered amongst the laboratory remains from Ringelhof (Switzerland), dated to the 13th century, although its provenance has not been ascertained (Kamber *et al.* 1998: 169). Even more, the spread of graphitic crucibles reaches across the ocean. Archaeological excavations at La Isabela (Dominican Republic) – America's first European town, founded by Columbus in 1493 – have uncovered 58 triangular crucibles. Most of them are very small and, in all likelihood, they were brought to the colony for assaying purposes. Their distribution throughout the site indicates that their use was generalised, rather than restricted to specialists (Deagan and Cruxent 1995: 5; 2002: 259-265). Thin-section petrographic analyses by David Killick (pers. comm. 2003) confirmed that the fabrics of these vessels are very rich in graphite inclusions. Even though the exact provenance of these crucibles has not been identified, their presence in America suggests that the reputation of the black crucibles from Central Europe may date back to at least the late 15th century.

Furthermore, over one hundred triangular crucibles of different sizes have been recovered from the Imperial Mint in Rio de Janeiro (Brazil), dated to the 18th or early 19th century. Most of them are very rich in graphite and bear stamps on their bases, very similar to the typically Obernzeller ones discussed above. These fall in three categories, each showing slight variations around the initials LK, PS and IS – perhaps the evolution of the marks during a relatively long temporal span. Alongside these marks there is another stamp with a number that increases with the size of the vessel. Nearly identical stamps are known from crucibles from other Brazilian sites. These crucibles must have been expensive and/or scarcely available, as they occur together with other vessels made of recycled graphitic sherds, crushed and mixed with non-graphitic clay, and probably manufactured locally. Interestingly, however, it seems that not all crucible bases were used in grog making: out of the 136 crucibles recovered in the mint, 87 constitute loose bases (Lima and da Silva 2003). Perhaps the bases were saved to be used as scorifiers or lids, or they were simply more difficult to crush – although the possibility of a preferential recovery of bases during the archaeological excavation cannot be discarded as an explanation.

Founded on a historical source mentioning the import of crucibles from Ips, Lima and da Silva (2003: 11) suggested that the purely graphitic crucibles came from this Bohemian producer. The extent of the trade of black crucibles from Central Europe

was thus exemplified, but new questions were posed for the present thesis. If these crucibles had truly been imported from Ips, why did they bear stamps so similar to the Obernzeller ones? To what extent is this stamp diagnostic of a certain provenance? If not provenance, what does this stamp tell us?

7.4. From prints through pots to becherovka: an enigmatic stamp

Let us start by recapitulating pertinent information. A range of crucible stamps, all of them analogous, have been used as a basis to ascribe the crucibles bearing them to Obernzell producers. In the cases of the crucibles found in Oxford and in Canterbury Barracks, this ascription seems unquestionable, since identical stamps are recorded in pottery known to be from Obernzell (figs. 125 and 129). For the other British examples discussed, no exact matches were identified during the author's study visit to the *Keramikmuseum* in Obernzell. Nonetheless, considering the strong similarities between these and some assured Obernzell stamps, and bearing in mind estimate figures of several thousands of stamps in the history of this village (R. Hammel pers. comm. 2003), it appears sensible to relate those vessels to the same production centre. The situation becomes more complicated when we come to discuss the crucibles found in Rio de Janeiro, where we find a strikingly similar stamp, but written information suggests a slightly different provenance. Moreover, even scientific analyses may fail to ascertain whether these crucibles were produced in Ips or in Obernzell, given that Ipsian crucible makers imported clay from Obernzell (Gaspari 1797: 591).

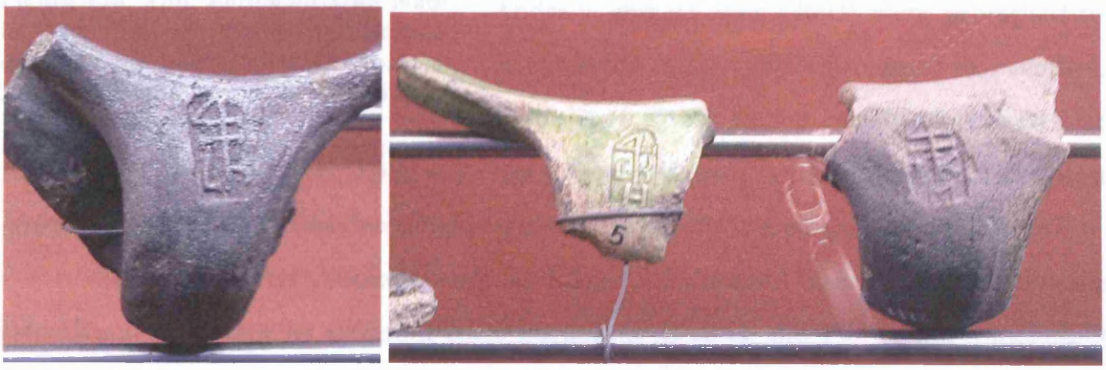


Figure 125. Some stamps in artefacts from Obernzell and exhibited in the Keramikmuseum. The left one is virtually identical to that of the crucible found in Oxford (see fig. 120).

Yet the quandary of this peculiar symbol may be confused further if we add another pot to the equation. The artefact shown in Figure 126 is a *Bartmannskrug* or 'Bearded Man Jug', a very popular late 16th-century salt-glazed stoneware type,

Figure 126. Top, *Bartmannskrug* made in Jan Emens' workshop. Bottom, detail of the house mark (Victoria & Albert Museum 764-1868, cf. Gaimster 1997: 236, No. 87).

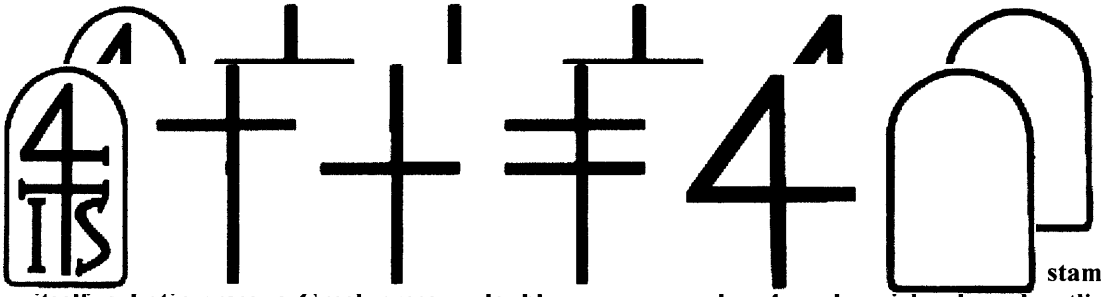
consisting of a round-bellied pitcher with a bearded face on its neck. The example illustrated here was made in the workshop of Jan Emens Mennicken (1540-1593), a famous pottery master who worked in Raeren (Belgium) (Gaimster 1997: 225, 236). A close look reveals that the mark in the central medallion bears a strong resemblance with those shown above, although with different initials: IEM (fig. 126). We therefore have a piece of pottery produced in a well demarcated context, arguably alien from Obernzell, yet sharing a similar stamp design. How can this be explained?

Two decades ago, Ingolf Bauer (1983: 36) briefly warned that the stamp based on a symbol resembling the number 4, normally with a second horizontal segment, was a relatively common type used by merchants in the early modern period. He also noted the presence of the same mark engraved on the wall of the west tower of the Obernzell castle, next to an annotation of the year 1573. More recently, Andrade and da Silva (2003) were prompted to research the origins and meaning of this symbol, which they found in the crucibles recovered at the mint in Rio de Janeiro. They were not acquainted with its occurrence in European pottery, however they considered the peculiar mark worth studying on its own merit.

As revealed by Andrade and da Silva, this symbol was indeed used with astonishing frequency by different trade corporations since the 15th century, including goldsmiths, engravers, sculptors, potters, glassmakers, painters, drawers, stonemasons and so forth. It appears with special incidence in printers and booksellers, with 70 occurrences in Paris and Lyon (France). Of special interest is its utilisation by John Siberch – the first printer to work in Cambridge – in his first book, published in 1521. The mark in this book is virtually identical, even in the initials (IS), to that found in

some of the crucibles recovered in Brazil, dated some two hundred and fifty years later. What is the reason behind such a wide span?

The symbol in question may be deconstructed in simpler shapes: apart from the characters, there is a Latin cross, a Greek cross, a double cross, a number 4 and, frequently, a well delineated niche (fig. 127). Each of them, and their combination, become meaningful in the context of Renaissance and later religion, hermetic chymistry and freemasonry. The double cross joins together the Greek cross – symbol of the macrocosmos –, and the Latin cross – the microcosmos –, in what seems a statement of the chymical theory of correspondence. In Jan Emens' mark, the two crosses are superimposed in different orientations, but the connotations could be the same. In the centre of the cross, the quintessence embodies the equilibrium amongst the four elements. For freemasons, this is a symbol of the Great Architect. With regards to the number 4, its interpretation is manifold: on the one hand, the addition of a segment may be a concealment of the cross – a subtle way of sharing religious values amongst initiated during the struggle of the Reformation, as done by 16th-century booksellers in Lyon; on the other hand, four is the totalising number: four are the elements, the humours, the cardinal points, the phases of the moon, the seasons... and, according to some sources, the stages of transmutation (cf. Lima and da Silva 2003).



stamp itself, a Latin cross, a Greek cross, a double cross, a number 4, and a niche-shaped outline (after Lima and da Silva 2003, modified).

Amongst 18th-century chymical symbology, variations of the number four and the double cross are used for a variety of elements, including crucibles, mercury, sulphur, salt, distillation or gold (fig. 128). It is worth noting that in Latin and in English, as in various other languages, the word for 'crucible' is etymologically rooted in the term 'cross' (cf. Lima and da Silva 2003).

Finally, the niche-shaped outline does not seem incidental, for it coincides with a symbol recurrent in masonic books and paraphernalia, representing the two columns and the vault of Solomon's House. As noted in section 4.3, the myth of this temple of

wisdom and perfection was the inspiring force for the construction of the Ashmolean Museum in Oxford but, more importantly, it also served as a foundation for freemasonry, its aims, secrecy, discipline and initiatic rites (Lima and da Silva 2003: 29-41 and 48).

Historically, masonic lodges appear to have emerged in the late Middle Ages. By the 18th century, when freemasonry enjoys a golden age, many have developed into hermetic and philosophical societies of men who seek individual and collective perfection. They give a fundamental importance to secrecy, which conveys the need of codes and symbols identifiable by the initiated only. The use of multiple icons that integrate several symbols is common, and these are frequently drawn from religion and alchemy. In sum, as contended by Andrade and da Silva (2003), it is very plausible that the enigmatic stamp is indeed a symbolic expression of the most precious masonic ideals, spread through the medium of material culture. The niche-shaped outline, which does not appear in earlier stamps, may have been an 18th-century addition. As to the slight variations seen in some of the stamps, such as the anchor ending at the base and other minor amendments, they could denote modifications made in successive generations of users of the same stamp, perhaps adding yet more meanings to the manifestly conceptual mark.

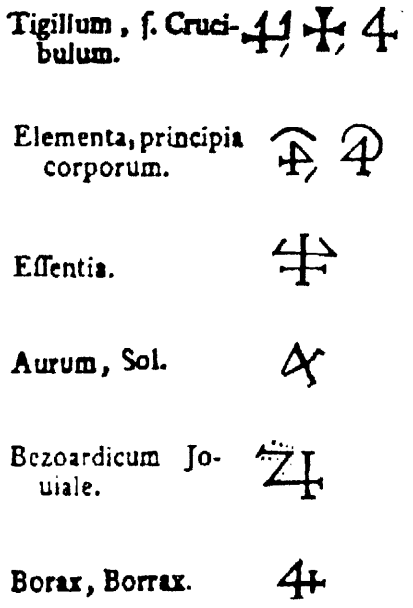


Figure 128. Some examples of chymical symbols combining a cross and the number four, from the *Medicinisch-Chymisch und Alchemistisches Oraculum*, published in Ulm in 1755[28].

The history of freemasonry is a complex topic beyond the scope of this thesis. More research into it may clarify when this symbol, probably existing before in the wider chymical domain, was incorporated into the imagery of freemasonry. This enigmatic stamp, however, raises another interesting and more relevant topic such as the transmission of religious or sectarian ideas by furnishing material culture – pottery in particular – with images taken from printed media. David Gaimster has investigated

[28] These images were obtained from Adam McLean's *Alchemy Website* <http://www.levity.com/alchemy/alchemical_symbols00.html> [accessed 14.07.2004], following a reference in Lima and da Silva (2003: 45).

how the development of woodblock printing and metal plate engraving in the Renaissance facilitated the swift spread of printed imagery across different social sectors of northern Europe, leading to a "democratisation in the consumption of imagery" (Gaimster 1997: 142, see also 37-40, 126-136 and 142-155; Gaimster 2003). During the 15th century, craftsmen incorporated some of this printed imagery in different media, including sculpture, medals, metal vessels, wooden furniture, domestic furnishings and pottery. The advance of mould technology in particular allowed the massive and relatively cheap production of ceramic objects displaying decorative motifs and more evocative images.

In the upheaval of the Reformation, religious and political images inspired by print media became recurrent in domestic objects. Stoneware potters applied to their productions moulded motifs representing biblical episodes, satirical caricatures, moralising scenes and political portraits and slogans (Gaimster 1997: 142-155). Thus ceramics became a more or less explicit channel for the communication of values, beliefs and bonds. As noted by Gaimster (1997: 150-151), the images utilised were probably the result of each potter's individual preferences, but they must have found a market of people from all social levels who wished to display their standpoints in the religious and political arena. As a matter of fact, the overt Protestant allegiance of some trade guilds led them to migrate to regions where they found more sympathy. Not only stoneware vessels, but also devotional pipeclay figurines and glazed earthenware stove-tiles, conveyed blatant political and religious messages, in the form of images taken from prints as well as from ecclesiastical altarpieces and statuary (Gaimster 2003). All of these are ubiquitous domestic artefacts which laid the celebration of personal beliefs at anyone's hands, even those hardly literate. When recovered by present-day archaeologists, they become hard evidence for a more accurate reconstruction of the extent and pace of Reformation (Gaimster 2003; 2004).

Figure 129. Stamps used by a corporation of crucible makers of Obernzell in the late 19th century (from Bauer 1983: 32). The central one appears in the graphitic crucible found in Canterbury Barracks (see fig. 124, p. 202).

The case of the stamp on the crucibles is peculiar. Firstly, it does not constitute moulded decoration on a domestic object but a house mark on a technical vessel. Secondly, considering its extremely wide distribution in terms of time, space and vehicles, as well as its relatively subtle semantics, the author wonders the extent to which this evocative symbol was comprehended by those who stamped it, or even those who consumed the goods where it was displayed. One may assume that a master crucible maker would originally be initiated in the meaning of his own house mark. However, would the potters working for him be aware of this? Allegedly, this would only be the case if they were initiated and thus shared the beliefs purported in the symbol. Furthermore, even if a 16th-century potter knew the secret of the symbol, perhaps his 19th-century descendant simply preserved the illustrious stamp but had forgotten the original meaning. This may have been the case in the Kaufmann family of Obernzell, likely the surname behind the letter K frequently found in these stamps: in the 17th century, Gabriel Kaufmann appears enjoying the privilege of supplying crucibles to the Imperial Mint (Bauer 1983: 31). Two centuries later, we find a likely descendant, Josef Kaufmann, associated with Gg. Saxinger and producing crucibles with remarkably similar stamps (fig. 129) [29]. Did the meaning of the symbol remain the same too?

Figure 130. Top, bottles of becherovka. Bottom, detail of the stamp printed in the label and also moulded in the glass (Pernod Ricard n.d.).

As an alternative hypothesis, was the presence of this particular symbol a response to customers' expectations? In other words, did potters add it to their products – perhaps

[29] Stephan (1995: 36, fig. 24) includes a drawing of a crucible showing a stamp very similar to this, and containing the initals IK, as probably originating from Hesse, another renowned crucible making region (see chapter 8). This attribution is in all likelihood erroneous.

unaware of its full meaning – simply because they sold more? It seems feasible that at least some manufacturers reproduced variations of this motif as imitations of other successful productions. Stemming from this, more questions may be asked: to what extent did the presence of the stamp condition the acquisition and use of crucibles? If it did at all, was it taken as a representation of appealing ideas or just as an indication of a renowned provenance?

To name but one appropriate example, the author was struck when noticing that the bottle of becherovka, the famous Czech spirit, still exhibits a house mark notably akin to that discussed here (fig. 130). The initials shown are those of Jan Becher (JB), the son of the early 19th-century pharmacist who started selling the beverage, even though the company now belongs to the multinational Pernod Ricard (Jan Becher - Pernod Ricard 2003). Is/was the becherovka producer a freemason? Does/did he know the historical meaning of the symbol? Do/did customers notice or mind the stamp?

Much information rests in pottery house marks, and research into their history and meanings is encouraged (cf. Gaimster 1997: 156-162). In 1431, a legal ordinance made it mandatory for black wares sold in the Vienna market to be stamped, and it is assumed that this would serve as an assurance for both potters' guilds and customers (Pittioni 1977: 94-95; Bauer 1983: 36). By the 19th century, some crucibles produced in Obernzell exhibited as many as six marks on their bases, whose origins and meanings are yet to be investigated in detail (Bauer 1976: 37). A few answers and many questions have been presented here, for which joint archaeological and historical work still has much to clarify.

7.5. Crucibles from Obernzell in Oberstockstall? Historical and archaeological information

Within the general framework of the production of black crucibles in Bavaria and surrounding regions, and having established the special role of Obernzell as a production centre, this chapter has to finish with a consideration of the provenance of the black crucibles found in Oberstockstall: were they produced in Obernzell?

This hypothesis seemed the most reasonable in a preliminary approach (von Osten 1998: 86). It is worth remembering that one of the chymists who arguably worked in Oberstockstall, Urban von Trenbach, was the archbishop of Passau between 1561 and 1598, a period overlapping with the functioning of the laboratory. As the ruler

of the archdiocese embracing Obernzell, he would have had easy access to the famous instruments produced in this village.

The study of the archaeological remains suggests otherwise, though. The analytical characterisation of the fabrics shows slight but potentially significant differences between the crucibles from Oberstockstall and the comparative samples from Obernzell and Oxford. Whilst all the unquestionably Obernzeller ceramic fabrics form a coherent group – both crucibles and ordinary ceramics –, the crucibles recovered in Oberstockstall diverge. As advanced above, this is noticeable in the chemical composition, with the Oberstockstall crucibles appearing richer in alumina and poorer in iron oxide (Table 16), as well as in the nature of the mineral inclusions, with the Oberstockstall samples showing more abundant zircon and scarcity of biotite and amphibole (see sections 6.2.1 and 7.3).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO	FeO
OBZL 01(crucible)	1.1	1.0	27.7	58.3	0.4	2.5	0.9	1.2	6.9
OX 1422 (crucible)	0.3	0.8	28.9	55.5	0.2	2.6	0.9	1.4	9.3
OB 362 (stamped)	0.2	1.0	27.1	59.0	0.2	1.7	1.0	1.4	8.3
OB Crucibles (n=6)	0.2	0.7	32.4	57.3	0.3	2.1	0.7	1.3	5.0

Table 16. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of (from top to bottom): the reference crucible from Obernzell, the Obernzeller crucible found in Oxford, a large pot found in Oberstockstall bearing an Obernzell stamp, and the crucibles found in Oberstockstall. Note the special divergence of the Oberstockstall crucibles, particularly in the alumina and iron oxide levels.

A further point of discrepancy is the stamp. It has been noted that a great array of stamps were used in Obernzell, and they have not been systematically catalogued. However, as far as it can be ascertained, the 'T stamp' seen in the Oberstockstall crucibles has not been recorded in any artefact excavated in Obernzell. Conversely, this stamp and variations of it appear, since the Middle Ages, in crucibles and other pottery spread across the region stretching along the Danube from Bavaria to Hungary, with a special concentration around Vienna (Holl 1976). It has been suggested that the T might stand for Tulln, a small village of pottery producers some 20 km from Vienna (Wiesinger 1937; Holl 1976; Pittioni 1976), but this possibility has not been thoroughly investigated yet.

In any case, the archaeological and analytical data contest the original assumption based on historical information and suggest an Austrian, rather than

Bavarian, provenance for the crucibles found in Oberstockstall. If this is the case for the crucibles, the same may be assumed for the scorifiers, which seemed to originate from the same potters (see section 6.3.3). This adds a further detail concerning the supply of specialised ceramics to the laboratory and, more generally, with regards to the diversity of the production of black crucibles in Renaissance Central Europe.

7.6. Summary

A combination of archaeological and historical sources documents the large-scale production of crucibles in Bavaria during the early modern period. The most important centre seems to have been in Obernzell, but other crucible producers were scattered through Bavaria, Bohemia and Austria. One of these satellite production nuclei was the town of Ips (Bohemia), which has, however, become historically more famous than Obernzell, despite the fact that the former imported clay from the latter.

The chronology and geographic distribution of these developments cannot be established more precisely without further archaeological research. At this stage, it is clear that, by the 16th century, the industry was well established. All the crucibles produced in this region seem to have shared a black lustre, which would have allowed their straightforward identification by the customer. Future research should try to clarify in more detail how the different producers of black crucibles related to each other, particularly in terms of their competition in the market.

The fabrics of these black crucibles are rich in alumina and do generally, but not invariably, contain graphite. As discussed in section 6.2, their resulting material properties and performance would have normally been very satisfactory. However, another point to be further elucidated is the selection of clays and the understanding of graphite. Possibly until the 18th century, naturally graphitic clays were exploited and contended for, but the connection between the mineral graphite and graphitic clays had not been fully realised. This interpretation, based on archaeological and archaeometric data, is supported by the fact that both entities – graphitic clays and graphite – were referred to with different, seemingly unrelated, names. During this period, it may be that greater importance was placed on the appearance of the wares as a factor conditioning their performance.

A further line for future research should involve a more systematic comparison between technical and ordinary ceramics. Contrary to traditional assumptions, the

samples analysed here indicate that graphitic clays were reserved for technical ceramics, both in the cases of Obernzell and Oberstockstall. It is the author's suspicion that the manufacture of ordinary pots with graphitic fabrics only became the norm in the 18th century, once it was realised that these could be made simply by mixing crushed graphite with any clay. From this moment, the availability of naturally graphitic clays would no longer have been such a decisive constraint.

Post-medieval crucibles almost certainly produced in Obernzell had been previously identified in Bavaria and Austria, but this thesis also allows their identification in England – namely in Oxford, London and Canterbury. Furthermore, black graphitic crucibles, quite possibly Bavarian, have also been found in the Dominican Republic and in Brazil. Even though many finds lack a clear context, the case studies presented here already document the use of graphitic crucibles in at least three different arenas: in chymical laboratories, in ore assaying contexts, and in a mint.

Variations of a complex stamp, primarily conspicuous in the use of a 4-shaped top, appear frequently on the bases of crucibles from Obernzell. However, similar stamps are observable in other pottery types produced in different centres, and in a variety of technological contexts and media from the 16th century until the present day. Therefore, the use of a stamp of this type as a provenancing feature should always be complemented with investigation of other parameters. It has been demonstrated that this stamp consists of several overlapping symbols representing a variety of ideas related to the worlds of chymistry and, especially, freemasonry. The evolving meanings and implications of this symbol, both from the perspective of makers and users, open another interesting path for future enquiry.

Finally, the comprehensive archaeological and analytical data challenge the original postulation as to the provenance of the crucibles found in the Oberstockstall laboratory. An Austrian source appears now more plausible than a Bavarian origin, and Obernzell in particular seems an unlikely source.

8. The bright crucibles from Hesse

"No object is mysterious. The mystery is in your eye."
Elisabeth Bowen (1899-1973)

8.1. Introduction: "the mystery of the Hessian wares"

In 1677, the chymist Robert Plot, who would become the first Professor of Chemistry at the University of Oxford, examined the English crucibles manufactured by John Dwight, long time potter, experimental chymist and entrepreneur. Plot concluded that Dwight "hath discovered also the mystery of the Hessian wares, and makes Vessels for retaining the penetrating Salts and Spirits of the Chymists, more serviceable than were ever made in England, or imported from Germany it self" (Plot 1677: 250). This secret arguably unveiled in Dwight's workshop was indeed a long standing enigma: since the late Middle Ages, crucibles manufactured in the region of Hesse (Central Germany) had been widely traded across the world, thus indicating their renowned quality.

More than three centuries after Plot's observation, in 1992, John Cotter recaptured the "mystery" to raise awareness of the "remarkably little attention paid to the subject of post-medieval crucibles" and, particularly, the triangular type (Cotter 1992: 256). On the basis of historical sources, Cotter contended that literally millions of these had been imported into Britain alone. Quite rightly, he noted that "perhaps the real 'mystery of the Hessian wares' is that so many were imported into this country [England] yet none can positively be identified" (Cotter 1992: 269). He also offered an account of written information relating to the production and trade of crucibles in post-medieval times, and a preliminary catalogue of archaeological examples.

The Hessian crucible has gone down in history as the best and the most famous. It probably is the only technical ceramic produced in medieval and modern Europe whose name and reputation transcend the scope of specialists' publications, to such an extent that the term 'Hessian' is often extended by metonym to refer to any triangular vessel. The reasons behind the high status of the Hessian crucibles appear to be both historical and technical, and can be traced back into the Middle Ages. An approach to

this topic will provide a counterpoint to the preceding chapter, while allowing a more comprehensive picture of the production, consumption and performance of crucibles in general. Like the previous one, this chapter will start by outlining the traditional knowledge about the Hessian wares, before completing it in the light of archaeological and archaeometric information deriving from the analyses of a range of crucibles recovered across the world. As a result, the foundations will be established for the resolution of both 'mysteries': on the one hand, the technical quality of the crucibles and why they were so highly esteemed; on the other hand, the clues to identify these vessels in archaeological contexts[30].

8.2. Traditional knowledge: historical and archaeological information

The history and archaeology of crucible production in the German region of Hesse has been addressed in detail, based on written documents and local archaeological finds (Stephan 1995), therefore only a brief outline, based on Stephan's book, is presented here. Even though the first written reference to the manufacture of crucibles dates to 1503, archaeological evidence attests the production of technical ceramics in the villages of Epterode and Almerode (later merged in 'Großalmerode') as early as the 12th century.

In the 17th century, local potters were in legal dispute with Count Moritz (1592-1627), himself a practitioner of chymistry, and holder of both the privileges for the exploitation of clays and the manufacture of crucibles. Count Moritz customarily leased these privileges to the highest bidder – on one occasion the leaseholders were not even German, but Dutch producers –, and this caused irritation amongst the locals. However, it is known that the illegal manufacture of crucibles took place to a considerable extent, before the final withdrawal of these privileges towards the end of the century (Stephan 1995: 12-16).

Historical sources relevant to the manufacture and trade of crucibles during the early stages of this industry are scanty. Only from the second quarter of the 18th century

[30] Given that the main focus of this work is laid on the crucibles themselves, rather than on the specific reactions carried out within, the discussion shall concentrate on the ceramic materials, giving details of the metallurgical or chemical residues when necessary, but without addressing the interpretation of their uses. For the crucibles from Jamestown and Burgsteinfurt, whose primary utilisations were less well known, a more thorough analytical approach was required. Relevant micrographs and numerical data are presented in Appendix 2, but the comprehensive discussion and interpretation are being prepared for publication separately, as they fall outside the scope of this thesis. For the other assemblages, previous scientific studies have been reported in chapter 4.

do we have direct references to the export of Hessian wares to England, Scandinavia, Russia, America, East India and China via the harbours of Bremen, Amsterdam and Danzig (Stephan 1995: 32). It is worth noting, however, the observation made by the English alchemist Thomas Norton when, in 1477, he praised the fire resistance of the crucibles "made of Stone", allegedly not produced "in any Country of English grounde" (Norton 1652: 95). This could constitute an early reference to the import of Hessian vessels.

An exceptionally vivid description of the crucible manufacturing process as it took place in the late 18th-century Hessian workshops may be obtained from a variety of sources (Beckmann 1787; Gatterer 1790; Funke 1800; all as cited in Stephan 1995). White or grey clay was dug out by the lease holders from particularly deep layers. This clay was mixed with fine sand in ratios varying from 1:1 to a more sandy 1:3 mix, and the paste was subsequently moistened and repeatedly kneaded by feet for several days. Interestingly, the sand was obtained from a specific location: the Fahren Creek, which was dammed up so that the sand could be collected from the sediment. Other sources mention the manufacture of crucibles with clay mixed with finely ground grog, in ratios ranging from 1:1.5 to 1:3[31].

According to these sources, triangular crucibles were made in six different size grades, which fitted into each other forming 'nests' that facilitated economic firing and transportation. Also beaker-shaped crucibles with one pouring lip were produced, as well as square ones – the latter, manufactured on demand only. All of them had normally flat, round bases, as they were thrown on the potter's wheel[32]. Purportedly, one single crucible maker could produce one thousand medium-sized crucibles in one day. It was crucial that the crucibles were thoroughly dried after the manufacture, for which they had to be placed in a draft. For this reason, crucible making was primarily a seasonal activity, and crucibles made during the humid winter were deemed of inferior quality. One source describes a kiln as elliptical and having a door on either side, which could be opened and closed to adjust the temperature. This must have been of a considerable size, as it took three to five men day's work to fill up a single kiln. The

[31] To counter possible confusions, it should be mentioned that present-day crucible makers in Großalmerode produce graphitic vessels, but this constitutes a 20th-century innovation.

[32] Although not mentioned in the 18th-century sources, Stephan (1995: 11, fig. 3) also shows triangular crucibles with a convex, pointy base, which would more easily sit within the uneven charcoal bed in a hearth, but would be inappropriate for the flat platform of an assay furnace. This crucible shape would also be convenient for operations such as stibnite parting (see section 2.2.2), where a very small metallic *regulus* was to be collected in the bottom of the vessel. Some of these crucibles were also seen by the author in the assemblage from the Porto Mint.

crucibles were packed quite tightly – as shown by archaeological examples of misfired nests with the crucibles permanently stuck to each other – and fired for 48 hours.

8.3. Further knowledge: archaeometry and comparative archaeology

8.3.1. Characterisation and distribution

Only detailed analytical studies of Hessian crucibles may help explain their technical quality, whilst providing clues for their identification. Before this work, however, archaeometric analyses of these vessels were scarce. Stephan (1995: 22-23) reported the elemental composition of a number of crucible samples, probably obtained by wet chemical analyses, as published in the 19th and 20th centuries. More recently, he presented bulk XRF analyses of two medieval Hessian crucibles used for glassmaking (Stephan 1998). These results were consistent in the high alumina levels of the paste, together with low concentrations of alkali and earth alkali elements. Nevertheless, the presumable degrees of precision and accuracy of these data do not permit reliable comparisons with other archaeological crucibles. In 1986, Freestone and Tite undertook a more systematic study of twenty crucible samples ranging from the Roman to the late medieval period, including the characterisation of clay matrices, temper and material properties. The information regarding at least one of the later crucibles presented by them, when compared to the data presented here, suggests that it could constitute a Hessian vessel, even though the lack of comparative data did not allow such attribution in the original study (Freestone and Tite 1986, see below)[33].

This section starts with the characterisation of some crucible fabrics from Großalmerode, the most important crucible production centre in the region of Hesse. From this, it will expand onto the analyses of samples from other sites across the world that, after this work, may be attributed to the same producer. These will add further details regarding the varying paste preparations and the spread of Hessian wares, both in terms of geographic span and specific contexts of utilisation. On this basis, the material properties and performance of the crucibles from Hesse will be discussed.

a) Großalmerode (Hesse, Germany)

[33] It may be worthwile remembering here that the SEM-EDS chemical compositions of crucibles presented in this thesis refer to the ceramic matrices, *i.e.* excluding discrete mineral inclusions, following the analytical approach developed by Freestone and Tite (1986), therefore our data are inter-comparable (see section 5.5).

The two crucible samples from Großalmerode are surface finds and their dates are unknown. Both sherds are thin (~5 mm) and only slightly curved, hence they may be body fragments of triangular crucibles. Their external appearance is similar to that of other Hessian crucibles published by Stephan: they are orange in colour, and their surface texture is grainy and pimply, occasionally showing striations from their manufacture on the potter's wheel. No obvious traces of use were noted (fig. 131).

The sample 52207V consists of an extremely fine and lean clay matrix containing about 30 vol% of quartz grains. These are subspherical and relatively small (≤500 μm), although rarely smaller than 100 μm, either because the temper was sorted or because the smallest particles have melted. All of them are shattered by thermal stress (figs. 132-133). The clay matrix shows continuous vitrification, with development of a network of fine voids. However, it appears very firm and is far from terminal distortion (fig. 134). Under XPL, the paste presents a peculiar aspect: the matrix is grey in colour, but the areas surrounding quartz grains are slightly less vitrified and show a bright orange colour, in spite of having exactly the same composition (figs. 135-136). Shrinkage cracks parallel to the body surfaces are abundant and generally preserve their angular shape. Apart from the quartz grains, the only remarkable inclusions are some iron rich minerals, which melted and fluxed the surrounding clay. These often appear as hollow subspherical inclusions surrounded by a dark, more vitrified, area, and occasionally erupt through the surface of the vessel (figs. 137-138). However, given their small size – normally ≤200 μm large – their presence does not seem to challenge the stability of the crucible. Finally, very rare occurrences of tiny zircon and other silicates were identified.

The other sample (Großalmerode 52205Z) is more altered by post-depositional processes, with corrosion products penetrating through the fabric cracks (fig. 139). Its microstructure is, however, noticeably akin to that of the previous sample. The clay matrix shows continuous vitrification, combining grey colours in the core with light orange to white areas around the quartz grains and on the surface. Sand grains are slightly less abundant (~20 vol%). They are very shattered and usually appear accompanied by shrinkage cracks (fig. 140).

The chemical composition of the clay matrices of both specimens is nearly identical. It reveals an outstanding refractoriness, with alumina concentrations around 37 wt% and the sum of the iron oxide, alkali and earth alkali oxides below 5 wt% (Table 17). The firing temperature of these vessels is estimated to have reached 1500 °C, *i.e.* above the usual temperatures at which they would be utilised. This is indicated by the

high degree of vitrification – despite the clay refractoriness –, and the fact that the surfaces of the smaller quartz grains appear buckling and bonding with the clay matrix. In addition, given the low iron oxide concentration of the paste (below 2 wt%), the bright orange colour of the surfaces denotes a very oxidising firing atmosphere.

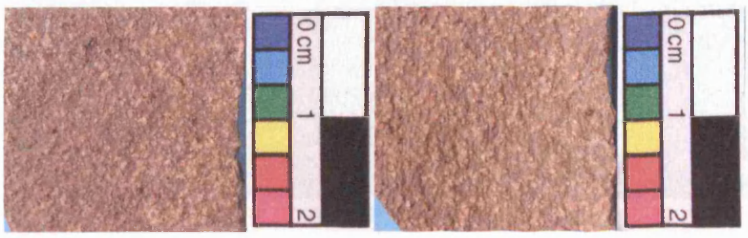


Figure 131. Detail of the surface of the crucibles from Großalmerode. Note the characteristic orange/ grey colour and the sandy surface (left, outer surface of Groß 52205Z; right, inner surface of Groß 52207V).

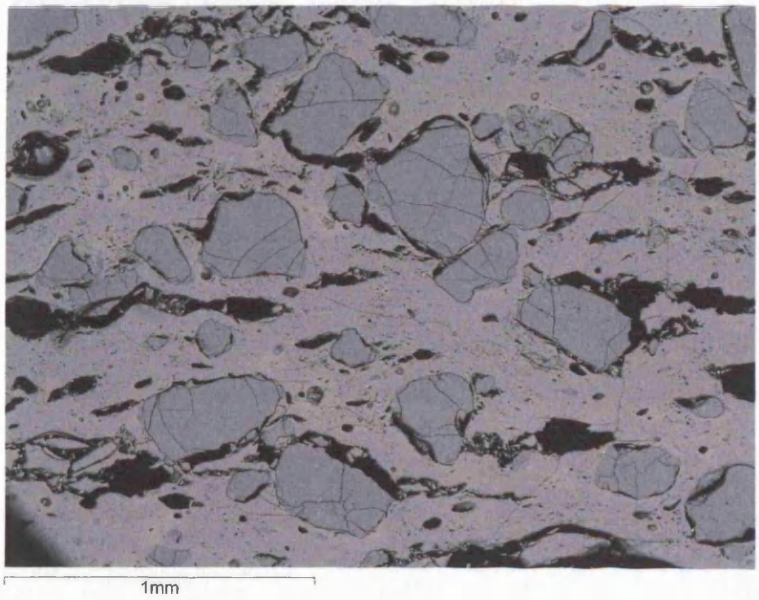


Figure 132. BSE photo-micrograph of the ceramic fabric of a Großalmerode crucible, showing abundant sub-spherical quartz grains, as well as shrinkage cracks (Groß 52207V/s1, 50x).

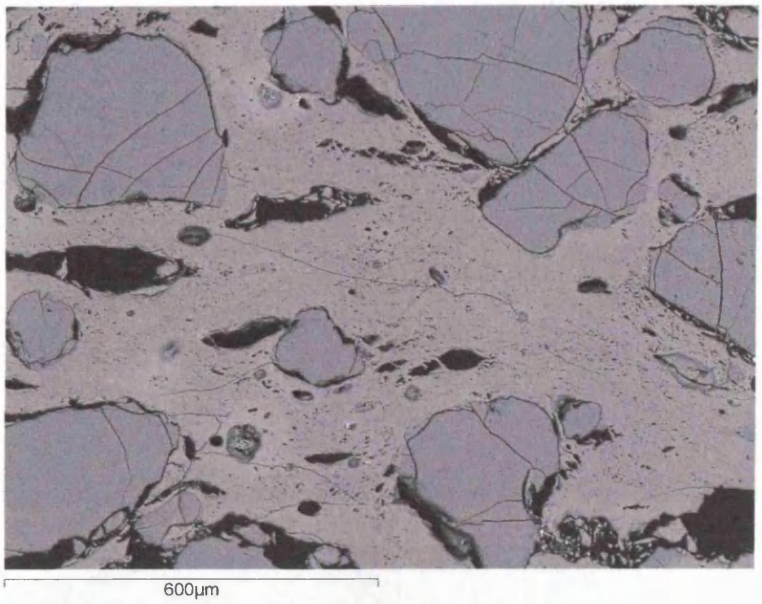


Figure 133. BSE photomicrograph of the same crucible fabric under higher magnification, showing the shattered condition of all the quartz grains (Groß 52207V/s1, 100x).

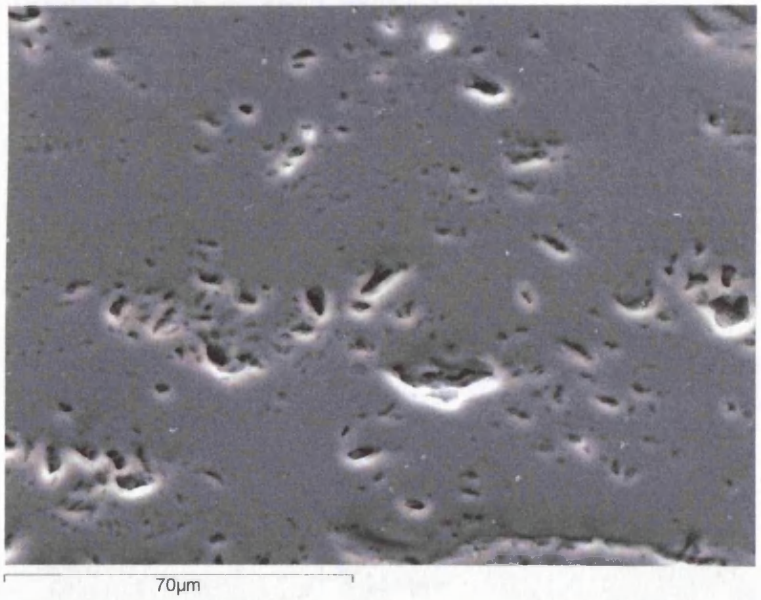


Figure 134. SE photomicrograph of the crucible matrix under very high magnification, showing continuous vitrification with fine voids (Groß 52207V/s1, 800x).

Figure 135. Fabric of the same crucible under XPL, showing a grey, vitrified matrix with orange areas preferentially around sand grains and cracks, as well as decomposing iron oxide minerals (dark spots) (Groß 52207V/s1, XPL, 50x, long axis represents ~2 mm).

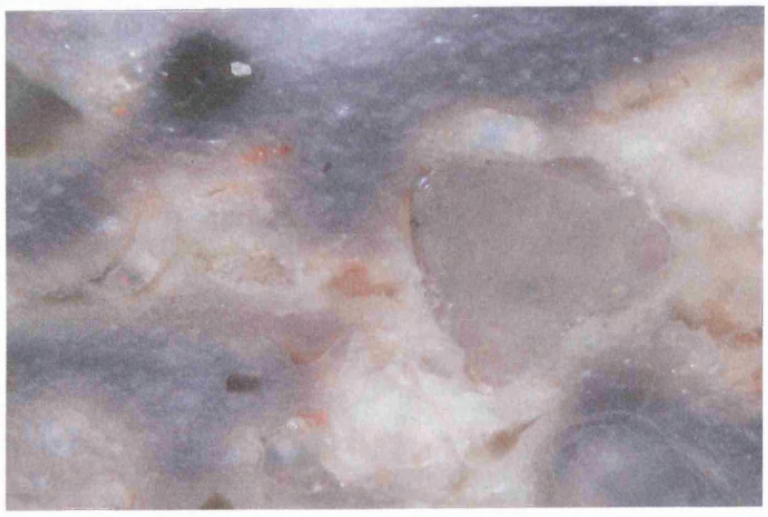


Figure 136. Detail of the same fabric (Groß 52207V/s1, XPL, 100x, long axis represents ~1 mm).

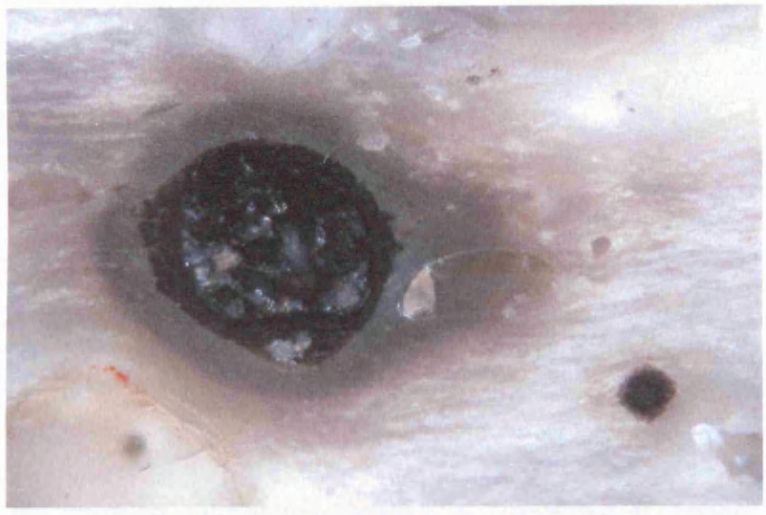


Figure 137. Detail of a decomposing iron oxide mineral, fluxing the surrounding fabric (Groß 52205Z/s1, XPL, 200x, long axis represents ~500 µm).

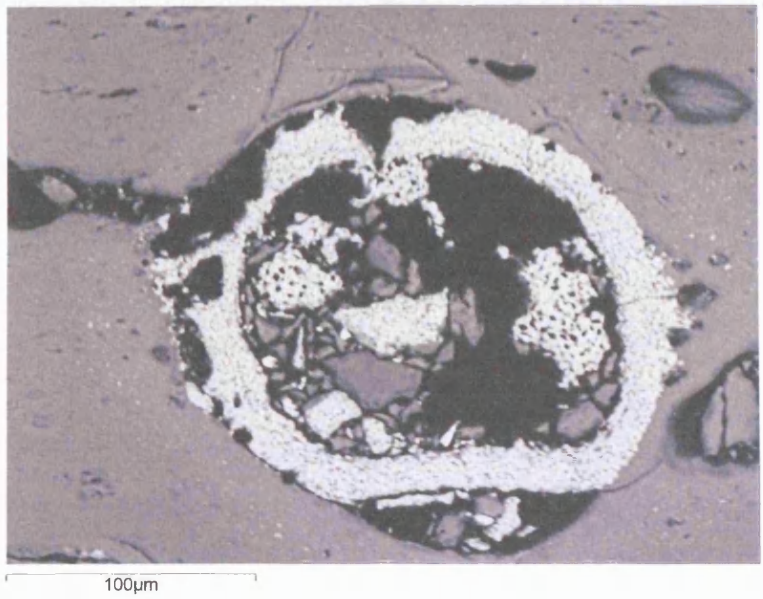


Figure 138. BSE photomicrograph of the same mineral. Note its hollow nature (Groß 52205Z/s1, 400x).

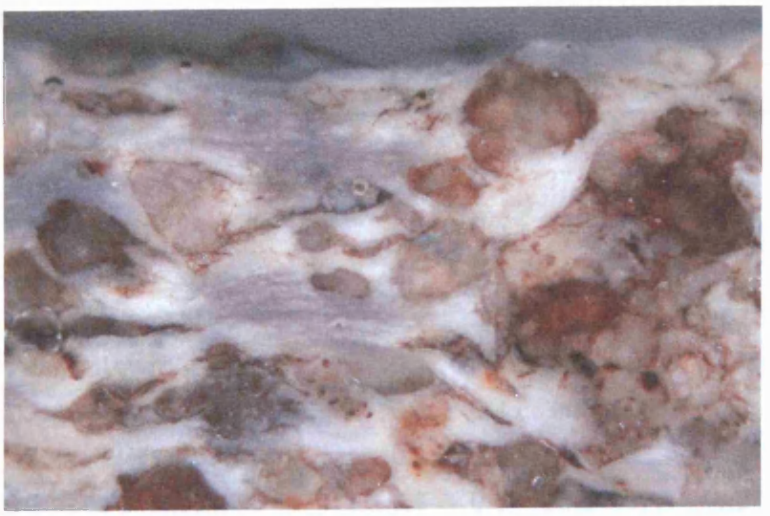


Figure 139. Fabric of a Großalmerode crucible. Note the darker colour of those areas of the matrix where no quartz grains are present, as well as the penetration of brown post-depositional products into the cracks (Groß 52205Z/s1, 50x, long axis represents ~2 mm).

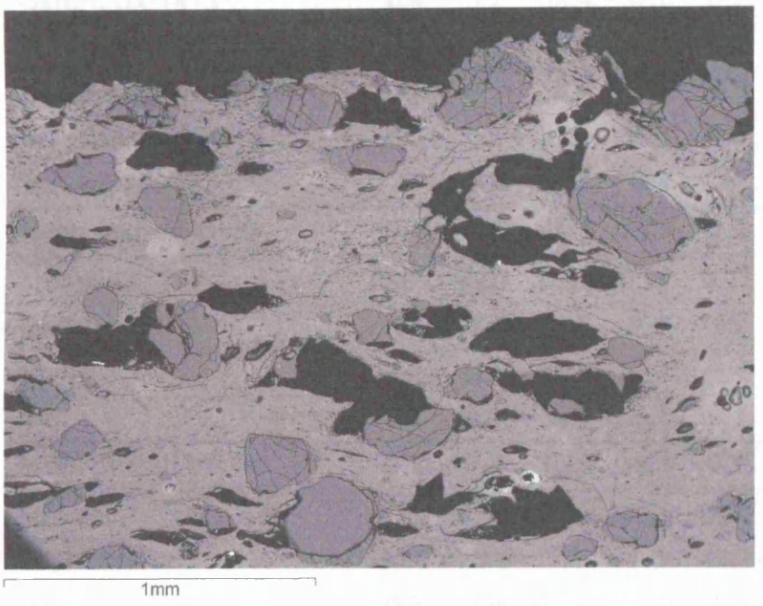


Figure 140. BSE photo-micrograph of the ceramic fabric of the Großalmerode crucible 52205Z, showing very shattered quartz grains, in slightly lower concentrations than 52207V (fig. 132), and a few bloated regions with molten minerals (brighter and with round voids) (Groß 52205Z/s1, 50x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
Großalmerode 52207V*	0.1	0.5	36.9	56.8	0.2	1.5	0.3	1.9	1.9
Großalmerode 52205Z*	0.1	0.5	37.5	56.5	0.4	1.2	0.3	1.9	1.7
Cripplegate WFG 18/129*	0.1	0.5	36.9	56.8	0.3	1.2	0.2	1.8	2.2
Cripplegate WFG 18/116	0.5	0.5	36.6	57.0	0.3	1.2	0.2	1.8	2.0
Jamestown JR 124F/C1	0.2	0.5	36.8	57.1	0.2	1.1	0.4	2.0	1.8
Jamestown A631/CC2	0.1	0.5	35.6	57.8	0.4	1.2	0.4	2.2	1.7
Jamestown JR 1024/CC3	0.2	0.4	36.8	56.4	0.3	1.5	0.2	1.9	2.3
Porto CI/91/3032/60*	0.2	0.7	36.6	56.9	0.1	1.4	0.3	1.8	2.0
Oxford OX n001*	0.2	0.5	36.5	57.1	0.2	1.4	0.2	2.0	2.0
Oxford OX 1413	0.4	0.2	36.4	58.0	0.2	1.9	0.2	1.6	1.2
Burgsteinfurt BGF 01	0.2	0.5	36.9	56.5	0.1	1.4	0.5	2.0	1.9
Burgsteinfurt BGF 02	0.1	0.5	36.4	57.2	0.3	1.7	0.4	2.0	1.6
Burgsteinfurt BGF 03	0.2	0.5	37.6	56.1	0.3	1.6	0.4	1.8	1.5
London 25842	-	0.8	37.7	56.2	-	1.2	0.4	1.9	1.4
Mean unused (n=5)	**0.1**	**0.5**	**36.9**	**56.8**	**0.2**	**1.3**	**0.3**	**1.9**	**2.0**
Std. dev.	0.05	0.09	0.39	0.22	0.11	0.13	0.05	0.08	0.18
Coeff. var.	50.0	18.0	1.1	0.4	55	10.0	16.7	4.2	9.0
Max.	0.2	0.7	37.5	57.1	0.4	1.5	0.3	2.0	2.2
Min.	0.1	0.5	36.5	56.5	0.1	1.2	0.2	1.8	1.7
Mean overall (n=14)	**0.2**	**0.5**	**36.8**	**56.9**	**0.3**	**1.4**	**0.3**	**1.9**	**1.8**
Std. dev.	0.12	0.13	0.55	0.55	0.10	0.23	0.1	0.14	0.31
Coeff. var.	60.0	26.0	1.5	1.0	33.3	16.4	33.3	7.4	17.2
Max.	0.5	0.8	37.7	58.0	0.4	1.9	0.5	2.2	2.3
Min.	0.1	0.2	35.6	56.1	0.1	1.1	0.2	1.6	1.2

Table 17. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of Hessian crucibles discussed in the text. The star (*) denotes those without clear traces of high-temperature utilisation. 'Mean unused' refers to these unused vessels, while 'Mean overall' refers to all of them. Data for London 25842 from Freestone and Tite (1986: 43).

b) Cripplegate Buildings (London, UK)

The crucibles from Cripplegate Buildings were deposited in the early 17th century. The archaeological context and the analytical study of the residues within the vessels suggested that they were used for activities related to goldsmithing and copper-alloy melting (Bayley 2003). Several fabric types were identified. Some of the crucibles showing fewer traces of use strongly resembled the reference samples from Großalmerode, particularly in the orange colour and the sandy texture, which suggested

that they could be Hessian. These were the thinnest vessels of the assemblage, with body walls as thin as 3 mm. Analytical examination of the triangular crucible WFG 18/129 (fig. 141) confirmed the original provenance hypothesis.

As in the samples characterised above, the clay matrix of this crucible shows extensive to continuous vitrification, and a neat colour difference between the generally grey matrix and the orange areas surrounding the quartz grains and at the surfaces (figs. 142-143). Its chemical composition is also strikingly analogous to that of other Hessian wares (Table 17). The fabric is tempered with ~20 vol% subspherical quartz grains, normally ≤500 μm large, and shows elongate shrinkage cracks. The smallest quartz inclusions seem to have dissolved within the paste. A number of other molten minerals were identified, relatively rich in iron and potash, which fluxed the surrounding paste. Under XPL, most of these bloated areas appear dark, but some of them, richer in potash and possibly constituting molten feldspars, are translucent (figs. 144-145).

Another specimen was removed from a used triangular crucible with a thicker wall and showing a lighter colour (WFG 18/116). The vessel contains a thin slag layer, very rich in soda and lime (each of them above 15 wt%), together with traces of lead, copper and zinc (fig. 146). Its fabric is tempered with ~30 vol% subspherical quartz grains (≤500 μm) and shows shrinkage cracks (fig. 147). The colour of the paste is generally light grey to white, probably due to its use in reducing conditions, although those regions without mineral inclusions appear slightly darker. The chemical composition of the paste and the nature of the temper clearly suggest that this crucible belongs to the same group as the previous one. In this fabric, however, most of the molten minerals identified constitute nodules rich in silica, alumina and iron oxide. One minuscule zircon inclusion was also noticed.

On this basis, the majority of the triangular crucibles from the assemblage may be ascribed to Hesse, although some exceptions, possibly local crucibles, are present as well (see section 9.3).

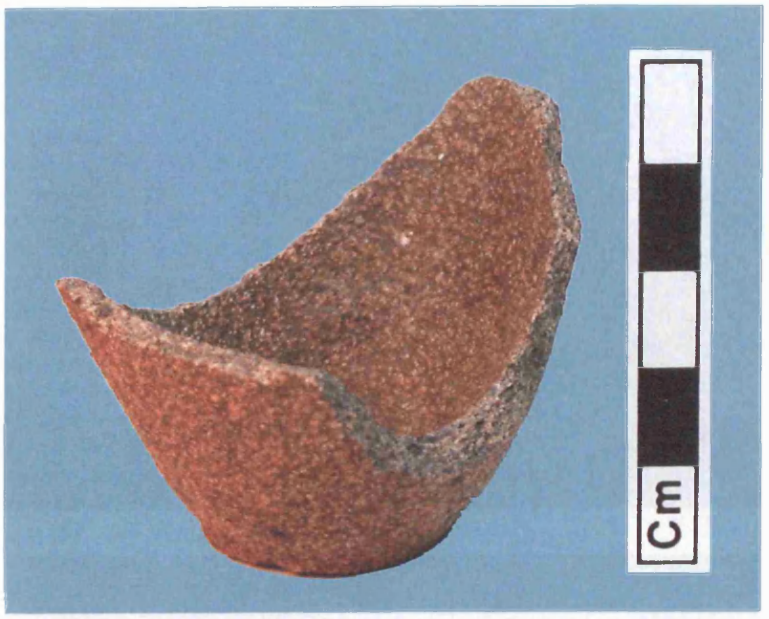


Figure 141. One of the Hessian crucibles identified in the Cripplegate assemblage (WFG 18/129).

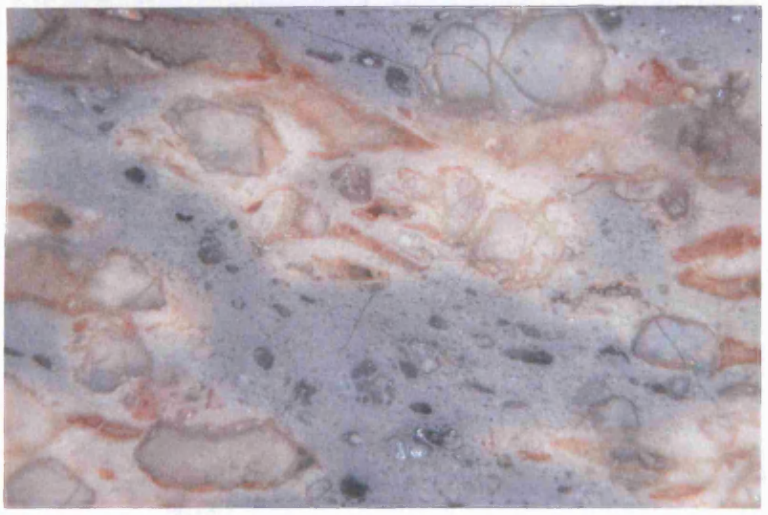


Figure 142. Ceramic fabric of the crucible shown in fig. 141. Note the more vitrified grey matrix and the orange regions surrounding quartz inclusions (WFG 18/129/s2, sXPL, 50x, long axis represents ~2 mm).

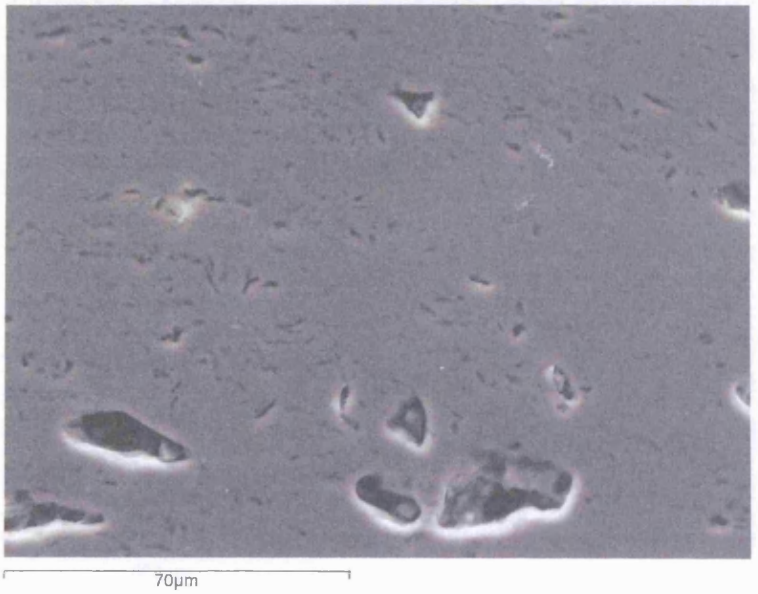


Figure 143. BSE photo-micrograph of the same crucible at high magnification, showing extensive vitrification with fine voids (WFG 18/129/s1, SE, 800x).

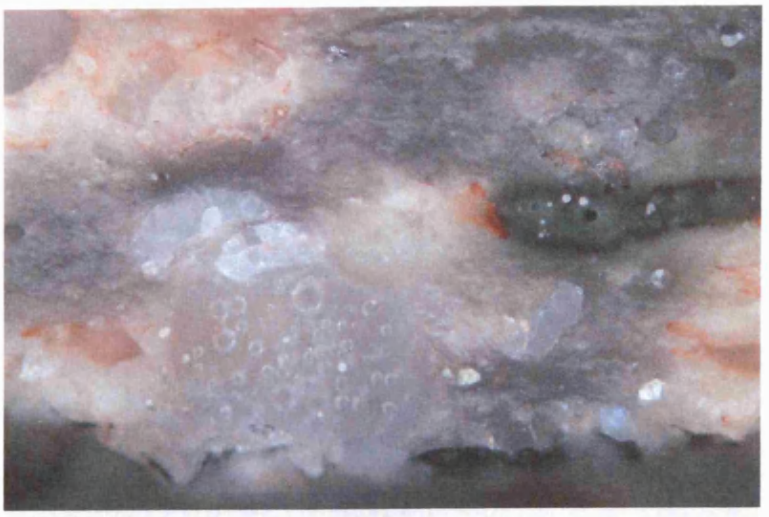


Figure 144. Section through the outer part of the Hessian crucible WFG 18/129, showing a molten feldspar grain (translucent, bottom), and a decomposing iron oxide (dark, right) (WFG 18/129/s2, XPL, 100x, long axis represents ~1 mm).

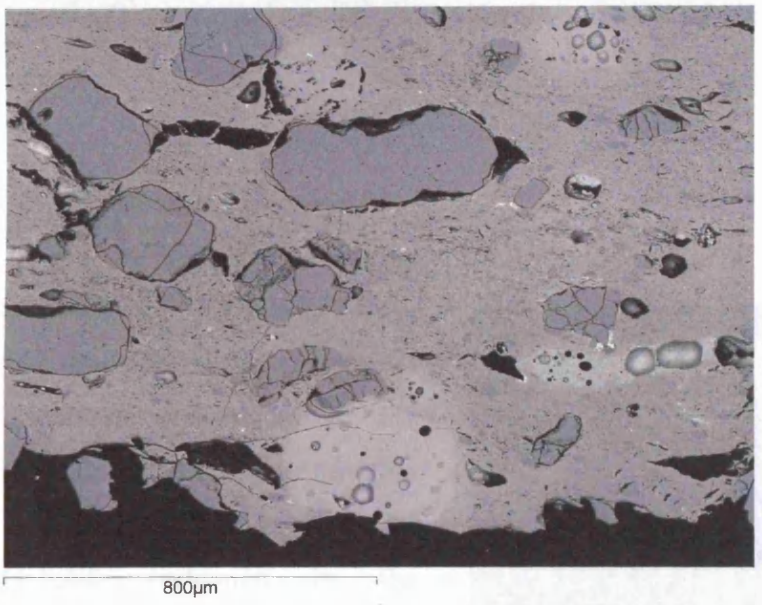


Figure 145. BSE photo-micrograph of the same area (WFG 18/129/s2, 75x).

Figure 146. Detail of the fabric of a crucible, showing the penetration of the alkali slag (bright) through shrinkage cracks (WFG 18/116/s1, BSE, 100x).

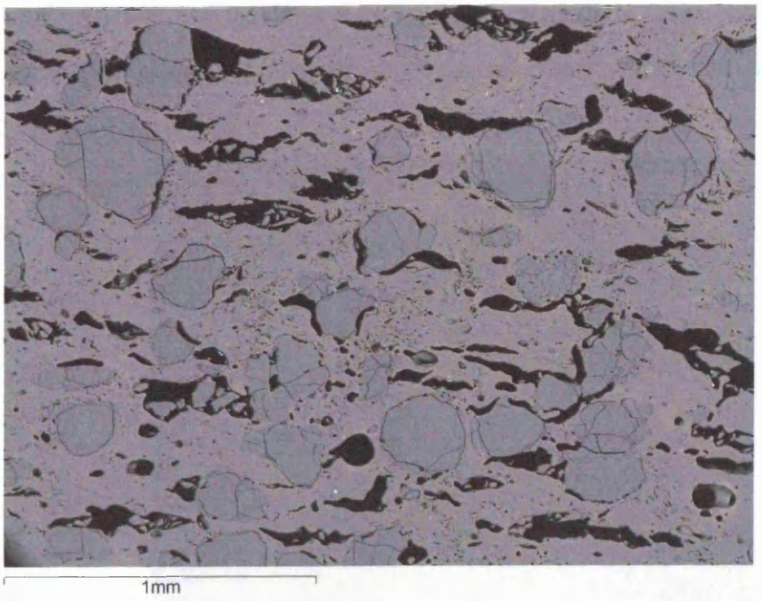


Figure 147. General view of the ceramic fabric of the same crucible, showing abundant subspherical quartz grains and shrinkage cracks (WFG 18/116/s1, BSE, 50x).

c) Jamestown (Virginia, USA)

The crucibles recovered in Jamestown (Virginia, USA), like those from the Cripplegate Buildings, were dated to the beginning of the 17th century (fig. 148). Given the involvement of German craftsmen in the Virginia Company, the original suspicion was that the technical ceramics might have been brought from Germany, namely from Hesse (Luccketti *et al.* 1994; Straube and Luccketti 1996). However, this hypothesis had not been confirmed until the present work. At least one of the preserved crucible bases shows two circular stamps containing two lines of letters, respectively

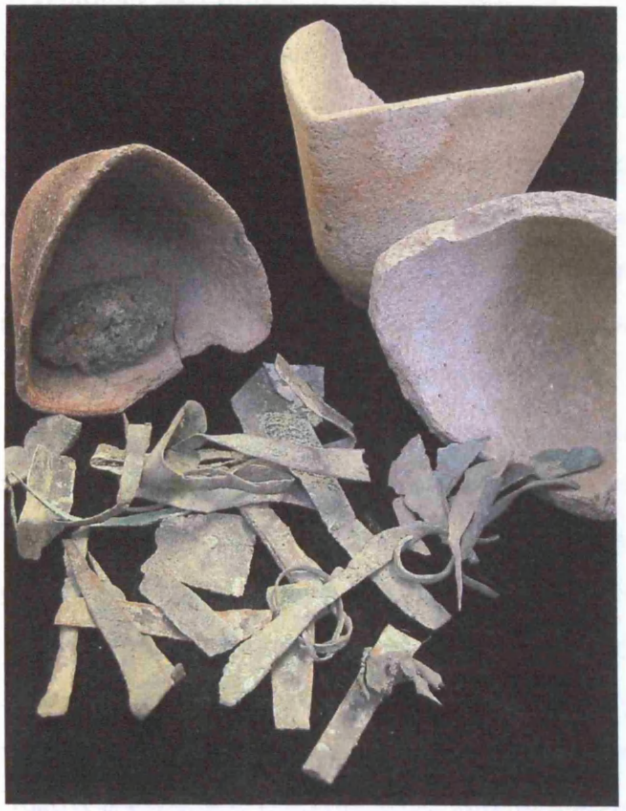


Figure 148. Group of crucibles and metallurgical remains from Jamestown (photo Carter Hudgins).

reading PTV and GER, separated by a central dot. This stamp, hitherto unidentified, is in fact identical to that observed in crucibles excavated in Hesse and published by Stephan (fig. 149).

Figure 149. Bottom of a crucible from Jamestown (photo Carter Hudgins). Inset, drawing of the same stamp, as identified by Stephan (1995: 36-37, figs. 24-25) in crucibles from Hesse.

The specimen JR 124F/C1 is a fragment of a beaker-shaped crucible (fig. 150). The paste appears white to light grey, except for the surface, which is looking darker and smoother as a result of contamination and vitrification during use. It is tempered with 30-40 vol% quartz grains, spheroid, with a low degree of angularity, and moderately well sorted – most of them between 200 and 500 μm large. In addition, there are some roundish argillaceous inclusions, about 1 mm large, which are compositionally identical to the main ceramic matrix and also contain quartz grains. These are generally surrounded by a shrinkage void, and their presence clearly causes a random crack distribution within the paste, compared to the parallel elongate cracks of most of the other crucibles. They are interpreted as grog fragments, although the high vitrification of the paste makes it difficult to confirm this (figs. 151-152). In spite of the thermal refractoriness indicated by the chemical analyses (see below), the ceramic matrix shows continuous vitrification, with development of a network of fine voids. This, together with the shattered condition of all the quartz inclusions, indicates a high firing temperature, either during original manufacture or during use. However, the paste appears very dense and firm. The clay matrix is extremely homogeneous, and no mineral inclusions other than the quartz grains were identified. Microscopic and SEM-EDS

analyses of the residues within the vessel indicate that it was used for melting a high-tin bronze with some lead and zinc (Appendix 2).

The crucible A 631/CC2 is a very small triangular vessel, with a flat base of 15 mm in diameter, possibly used for the assay of pyrites for gold (fig. 153). It also exhibits surface contamination and evidence of exposition to very high temperatures, although it does not appear to have become unstable. The clay matrix is grey and shows development of fine to medium vitrification voids (fig. 154). All the quartz inclusions (in this case, ~20 vol%) are shattered. There are relatively big elongate cracks across the body wall, with their shapes slightly buckling as a result of the high temperature. As in the previous sample, the paste is very lean and homogeneous – only some rutile and one ilmenite inclusions were identified as discrete minerals, all of them minuscule. Also a few bloated areas are present, up to 1 mm large, dark in XPL, submetallic in PL. SEM-EDS indicated high concentrations of iron oxide in these areas, as well as a higher alumina to silica ratio than in the ceramic matrix (fig. 155).

Finally, JR 1024/CC3 is a fragment of a relatively big, beaker-shaped crucible, which might also have been used for an assaying reaction (figs. 156-157). Its surface appears smooth and lustrous due to vitrification and contamination during use, but its microstructure is analogous to that of the previous specimen, albeit with a larger concentration of quartz temper (~30 vol%) (fig. 157).

The chemical composition of the ceramic matrices of the three crucibles show slightly higher variation ranges when compared to the reference samples, partly a result of varying contamination from use. However, they can reasonably be ascribed to the same group (Table 17, p. 223).

Figure 150. Crucible fragment JR 124F/C1.

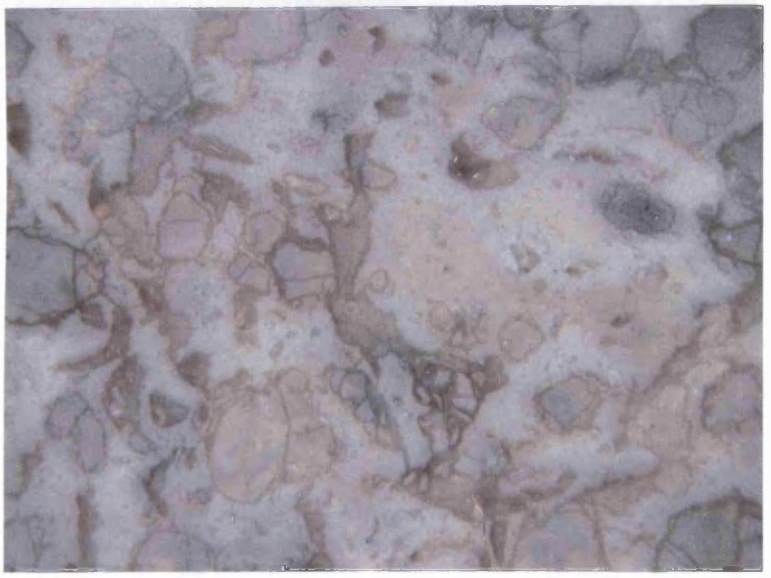


Figure 151. Detail of the fabric of the crucible, showing an argillaceous inclusion surrounded by a shrinkage crack (right), which is interpreted as grog (JR 124F/C1/s1, sXPL, 50x, long axis represents ~2 mm).

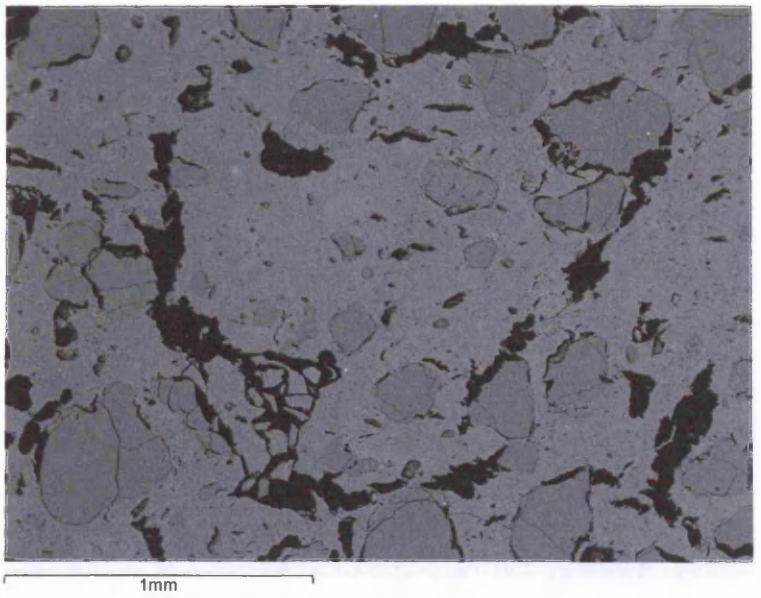


Figure 152. BSE photomicrograph of the same fabric area (JR 124F/C1/s1, 50x).

Figure 153. Crucible sample A631/CC2/631.

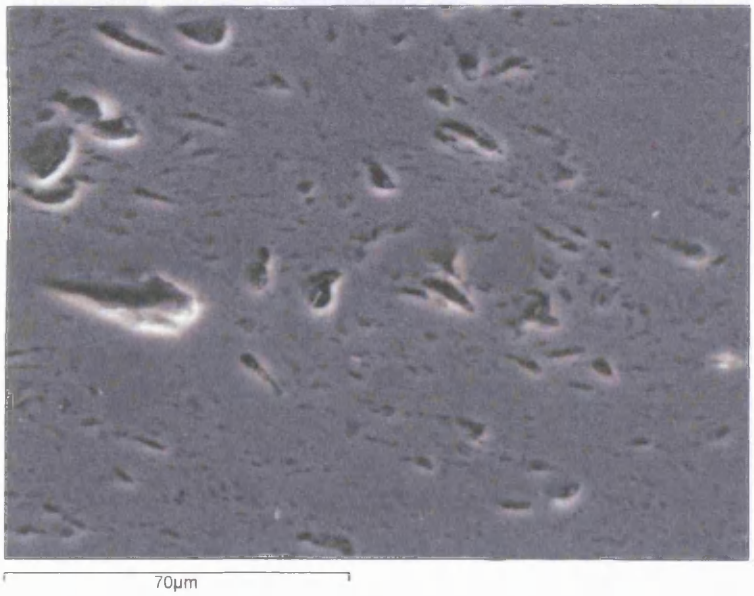


Figure 154. Detail of the fabric of the crucible, showing complete vitrification. Note that larger voids are not shown in the image (A631/CC2/631/s1, SE, 800x).

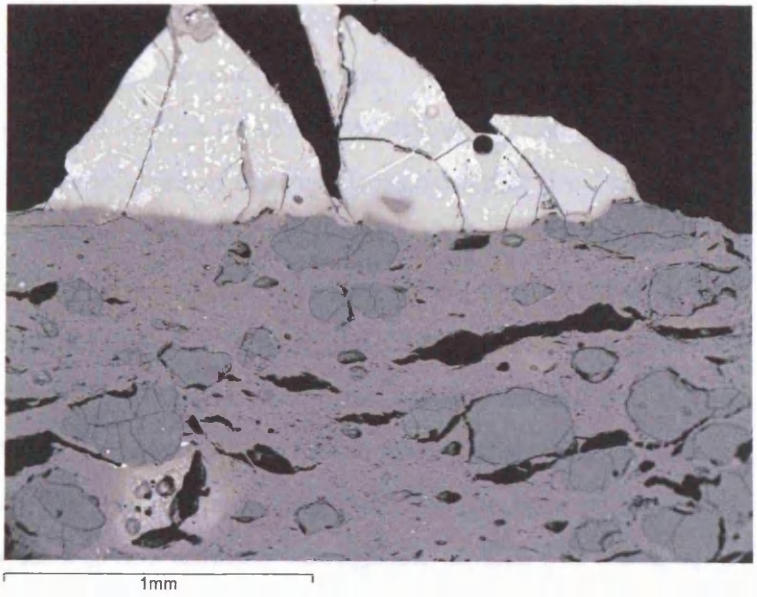


Figure 155. Section through the same crucible, showing the quartz-tempered fabric with one small bloated region (bottom) and slag remains adhering to the inner surface (top) (A631/CC2/631/s1, BSE, 50x).

Figure 156. Crucible rim fragment JR 1024/CC3. Note the shiny glaze and relatively smooth surface resulting from contamination and vitrification during use.

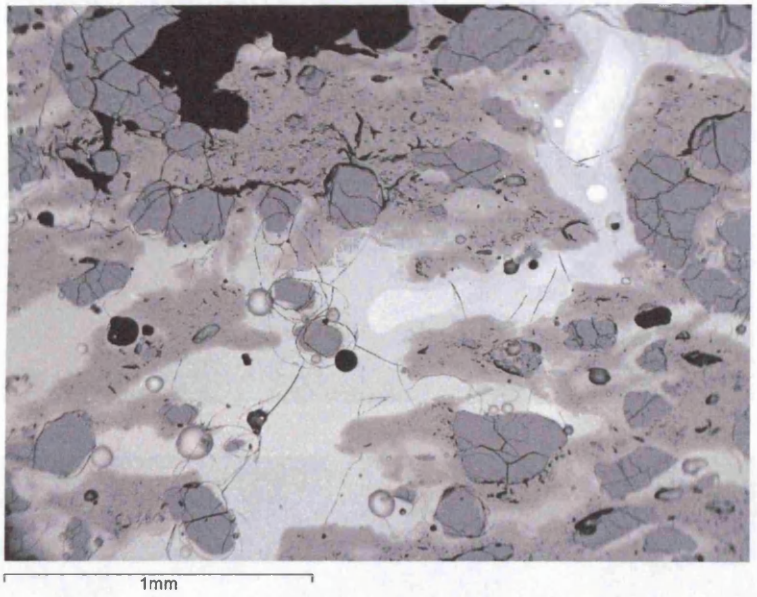


Figure 157. BSE photomicrograph of the crucible JR 1024/CC3, showing the penetration of slag, very rich in lime and copper oxide, through the fabric cracks (JR 1024/CC3/s1, 50x).

d) Porto Mint (Portugal)

The specimen from the Porto Mint (Portugal) was removed from a small unused triangular crucible, notably similar to some of the vessels recovered at the Cripplegate Buildings (fig. 158). Unfortunately, it lacks a clear stratigraphic context, therefore it may date to either of the two periods of activity in the mint, the first one between the 14th and the 16th centuries, and the second one in the late 18th century. Its texture is particularly sandy, especially on the inner surface, as a result of the thin walls (3 mm) and the abundant rounded quartz grains erupting through the surface (40 vol%, up to 1 mm large) (fig. 159). The surface colour is orange, but closer examination of the fabric reveals the typical

Figure 158. Fragment of the crucible CI/91/3032/60 from the Porto Mint, showing the characteristic appearance of the Hessian wares.

combination of a highly vitrified grey matrix with lighter grey or orange areas surrounding the temper. The paste contains numerous but minuscule black spots – bright in BSE images –, created by decomposing iron oxide inclusions (fig. 160). Chemically, the clay matrix falls within the same range as all the other Hessian wares (Table 17, p. 223).

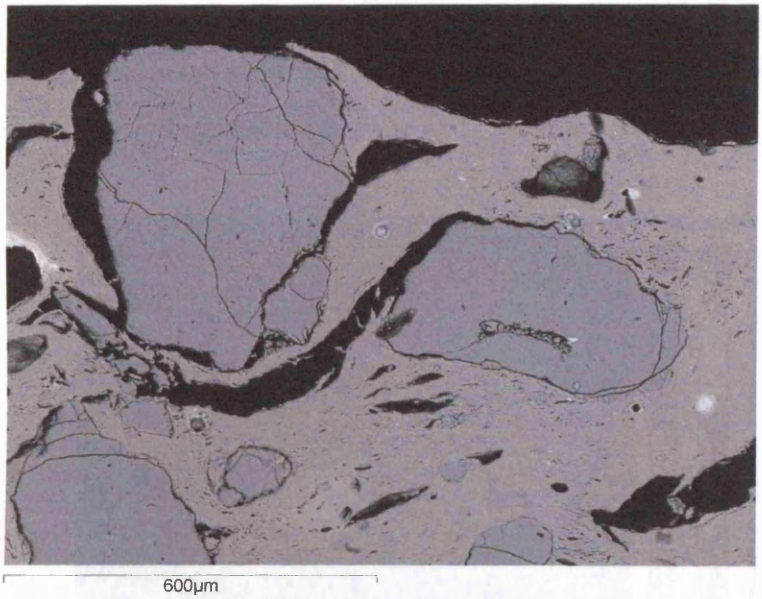


Figure 159. Detail of a sand grain erupting through the inner surface of a crucible (Porto CI/91/3032/60/s1, BSE, 100x).

Figure 160. Fabric of the same crucible, showing quartz temper, shrinkage cracks, and very abundant minuscule iron minerals (dark spots) (Porto CI/91/3032/60/s1, XPL, 50x).

e) Old Ashmolean Laboratory (Oxford, UK)

Alongside the graphitic crucible discussed in the previous chapter (section 7.3.2), two other crucible fragments from the Old Ashmolean were analysed. One of them is an orange, seemingly unused, triangular crucible, each side of the rim being 50 mm long; the other one is a smaller, beaker-shaped vessel, 45 mm high, with a rimsherd of

another (or the same?) crucible stuck to the outer surface and clear evidence of high-temperature use.

The analysis of the triangular crucible (OX n001; figs. 161-162) confirmed the hypothesis of a Hessian provenance, as its paste microstructure is analogous to that of the other unused crucibles from Hesse. It is tempered with 30-40 vol% subspherical quartz grains, up to 1 mm large, which appear shattered by thermal stress and frequently erupt through the surface forming bright orange specks. Many tiny decomposing black iron oxides were identified, together with a few larger (≤500 μm), translucent, melting feldspars, and minuscule ilmenite and rutile (figs. 163-164).

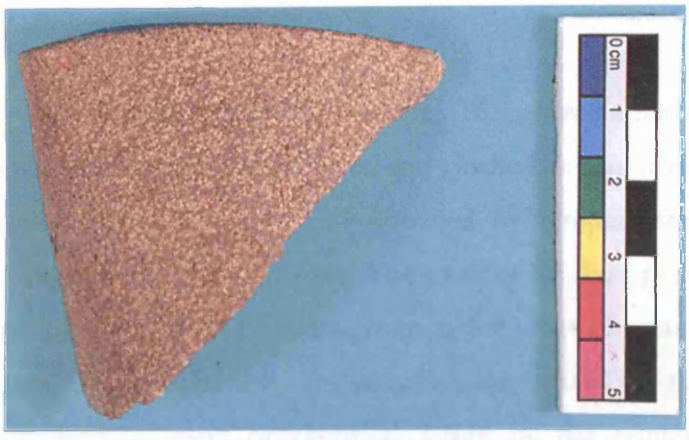


Figure 161. Triangular crucible fragment OX n001.

Figure 162. Detail of the surface of the same crucible, showing the typically Hessian colour and texture.

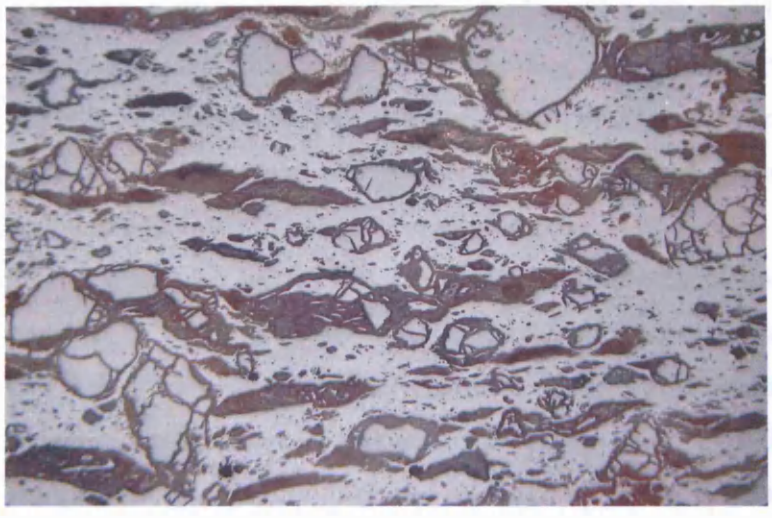


Figure 163. Fabric of the crucible OX n001, showing subspherical quartz temper and abundant shrinkage cracks oriented parallel to the surfaces (OX n001, PL, 50x, long axis represents ~2 mm).

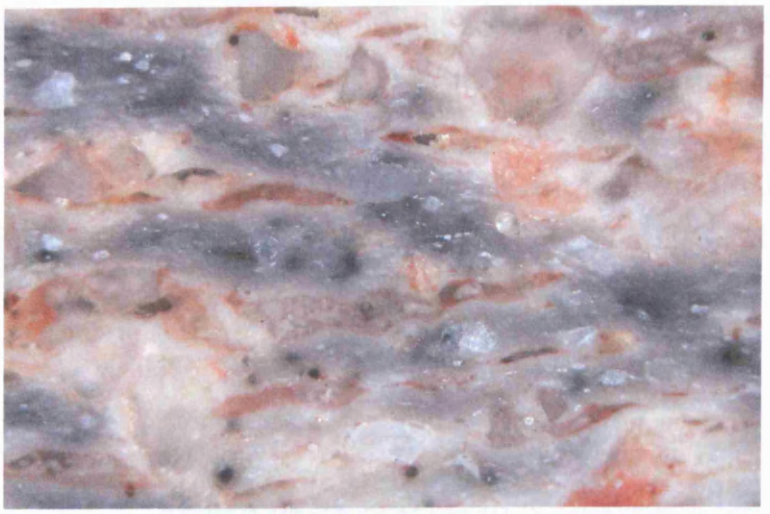


Figure 164. Another view of the same area of the fabric. Note the orange regions around the larger quartz inclusions, as well as the presence of small decomposing minerals (OX n001, XPL, 50x, long axis represents ~2 mm).

The other crucible (OX 1413; fig. 165) shows a darker brown surface, also very sandy but much more contaminated and vitrified. It was selected because of its similarity to another vessel that appeared distorted by temperature, which might suggest an inferior heat resistance (fig. 166). The analysis of this specimen shows, however, that this crucible belongs to the same group as the other Hessian ones, and is therefore very refractory (Table 17, p. 223). The actual reason behind the failure of some of the vessels is more likely a result of chemical attack by the content and an extremely high temperature of use. As noted by Chris Salter (Hull 2003: 11-13; Bennett *et al.* 2000: 31-47), this and other crucibles from the assemblage underwent exceptionally high temperatures, certainly higher than necessary for melting the substances held within. In this case, the clay matrix shows complete vitrification characterised by massive glass formation with medium to coarse subspherical voids (fig. 167). All the quartz grains, which are cracked throughout, seem buckling and dissolving within the clay matrix. The glassy layer adhering to the inner surface and penetrating into the fabric is composed of molten ceramic material with a strong contribution of manganese oxide (25 wt%) and containing globules of manganese sulphide (fig. 168).

While the aim of this thesis is not to investigate the specific utilisation of the crucibles, it is worth noting how these observations match the known rationale of the Old Ashmolean laboratory, for chemical research and teaching, with experiments on a potentially wide range of materials, including the then unknown manganese and its compounds. On the contrary, the scientific approach disproves the original suggestion regarding the provenance of the crucibles, based on the historical evidence for the

relationship between John Dwight and Robert Plot. Whilst Graham Hull (2003: 24) concluded that "it is highly likely that [...] the tall triangular crucibles [...] were of Dwight's manufacture and may even pre-date the earliest known English made examples", the analytical study indicates that the vessels found in the Old Ashmolean were imported from different centres on the continent (see also section 7.3.2).

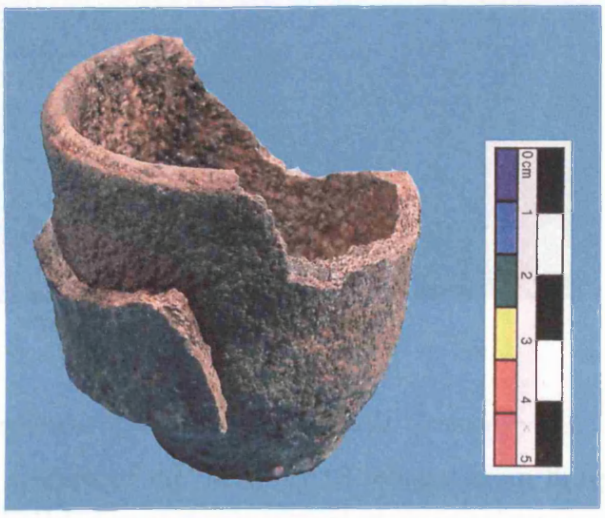


Figure 165. Small crucible from the Old Ashmolean laboratory. Note the rim sherd adhering to its outer surface (OX 1413).

Figure 166. Top view of two similar crucibles from the same assemblage. Note the clear distortion of the left one (OX 1406 and OX 1405).

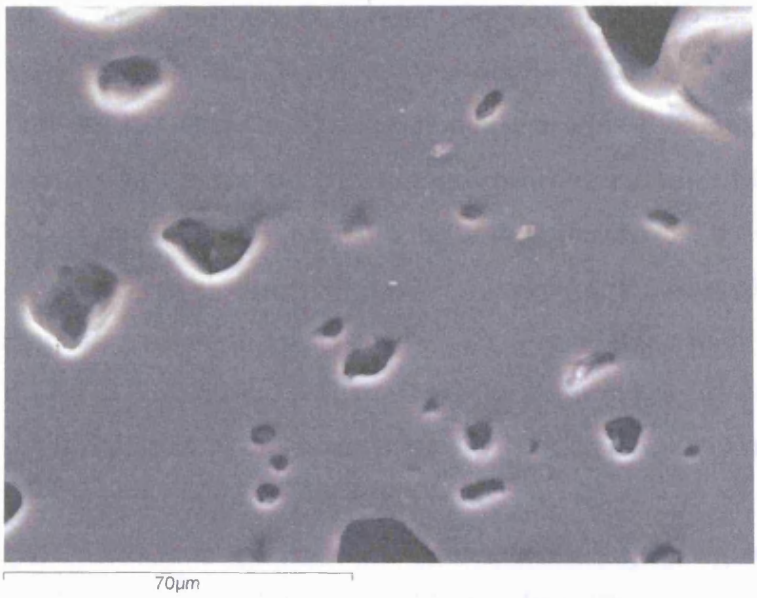


Figure 167. Detail of one crucible fabric under high magnification, showing complete vitrification and development of medium to coarse voids (OX 1413/s1, 800x).

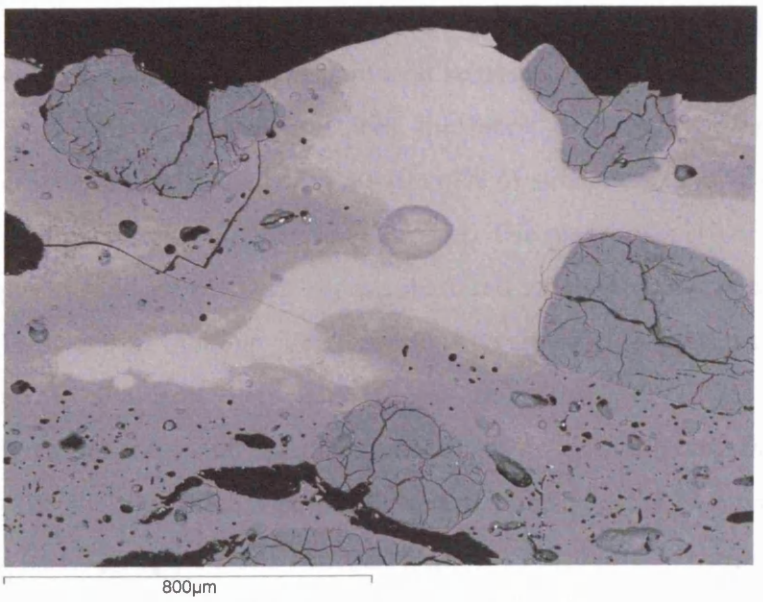


Figure 168. Detail of the slag penetrating from the inner surface through cracks and softening the paste. Note the severe cracking and the buckling of the quartz grains (OX 1413/s1, BSE, 75x).

f) Burgsteinfurt (North Rhine-Westphalia, Germany)

The analysis of the metallurgical remains from Burgsteinfurt suggests that they originate from a workshop where different copper alloys were melted and cast. Of particular interest is the presence of one crucible that was presumably used for 'speltering', *i.e.* the production of high-zinc brass by co-fusion of copper with pure metallic zinc (see Appendix 2). Given the uncertainties regarding the introduction of this technique in Europe, it is particularly unfortunate that no clear archaeological context or dates are known for this assemblage. Nevertheless, the characterisation of the crucible fabrics serves to broaden our knowledge of the Hessian wares.

The four crucibles from Burgsteinfurt have thick, flat, round bases and triangular rims. They represent three different size ranges. The first one (BGF 03 and BGF 04) has a base diameter of 37 mm, a height of 98 mm, and a thin body (4-5 mm). The side of the rim is 75 mm long. In the second one (BGF 02), the base is slightly bigger (40 mm in diameter) and the body is thicker (5-7 mm), but the height cannot be reconstructed. Finally, BGF 01 is the biggest type, although not the thickest. It has a base diameter of 50 mm, a height of 128 mm, and a body thickness of 5-6 mm, only thicker at the base and the rim. From the preserved fragments, the original length of the rim is estimated to be 85 mm. Overall, these crucibles appear much slimmer than those recovered in Oberstockstall (fig. 169).

The external surface of these vessels generally shows a smooth layer of vitrification in a dark brown to black colour. This layer, as analysed by SEM-EDS in

BGF 03/s1, showed exceptionally high iron oxide concentrations, in the range of 20 wt%, as well as some crusts of iron sulphide.

Except for the surfaces, the fabric of these crucibles is light grey to white in colour. It is tempered with 30-40 vol% of subspherical sand grains, not very well sorted and reaching up to 1 mm in diameter. The great majority of these inclusions are quartz grains, and all of them appear shattered as a result of thermal stress. The smallest of these appear melting. The paste shows very large shrinkage cracks along the body, some up to 0.5 mm wide and several millimetres long. The matrix is very lean and homogeneous, and it is compositionally very similar to the other Hessian crucibles (Table 17, p. 223). It is heavily vitrified and appears as a continuous glass phase (figs. 170-173). Apart from the quartz and some molten feldspar grains, the only discrete mineral inclusions are rare occurrences of rutile.

Figure 169. Comparison between one of the crucibles recovered in Oberstockstall (left) and one of the vessels found in Burgsteinfurt (right), where the slenderness of the latter may be noticed. Note the deep black surface colour and relatively smooth appearance of the latter, in spite of its Hessian quality (OB 307 and BGF 04).

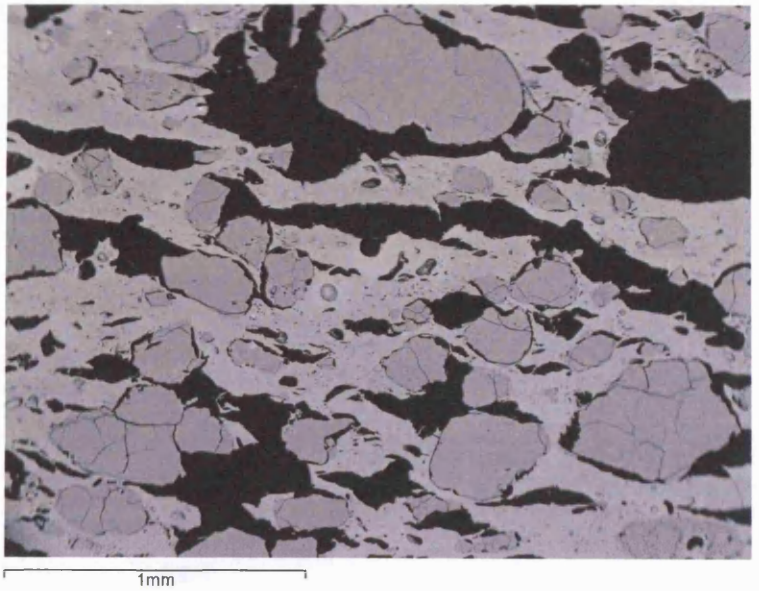


Figure 170. Fabric of the crucible BGF 02, showing abundant quartz temper and shrinkage cracks (BGF 02/s1, BSE, 50x).

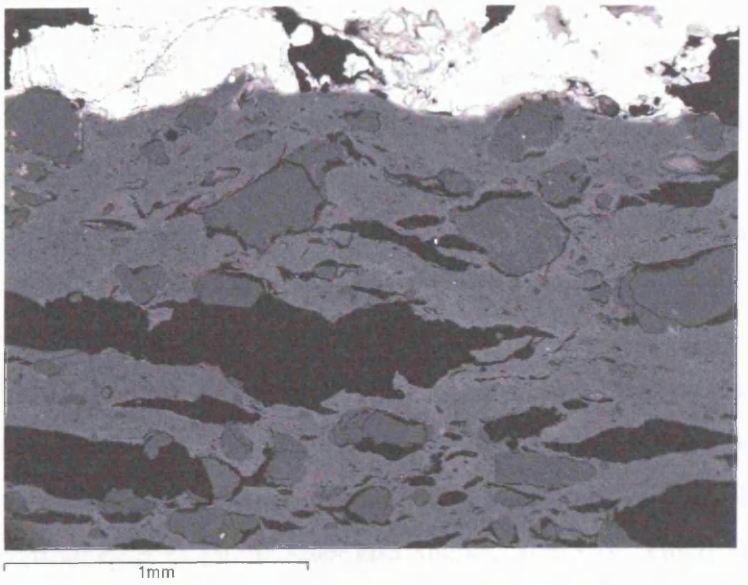


Figure 171. Detail of the crucible BGF 01. Note the substantial zinc oxide-rich crust adhering to the inner surface (top), as well as the large shrinkage cracks within the paste (BGF 01/s1, BSE, 50x).

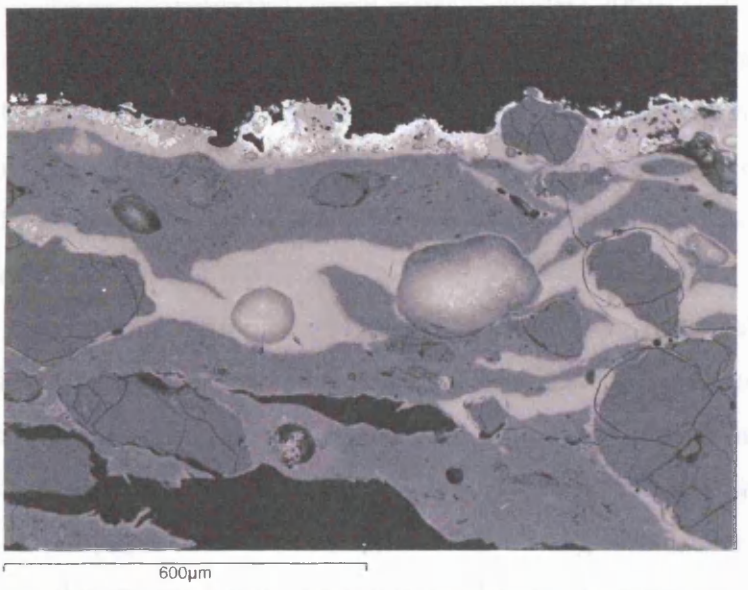


Figure 172. Detail of the slag within crucible BGF 02, penetrating into the fabric through cracks (BGF 02/s1, BSE, 100x).

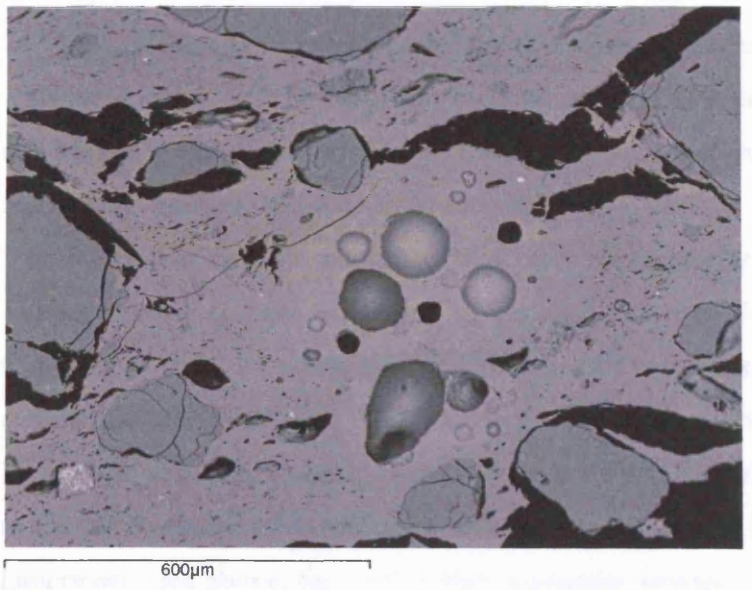


Figure 173. Detail of a molten mineral within the fabric of a crucible, probably a feldspar (BGF 02/s1, BSE, 100x).

g) Other archaeological examples of Hessian crucibles

On the basis of their fabric appearance and, in some cases, their stamps, some archaeological crucibles may be tentatively ascribed to Hesse, despite the lack of conclusive analytical data and the fact that, in each case, the authors who published the crucibles were unable to identify their provenance.

As far as the author is aware, the earliest examples of Hessian crucibles known outside Central Europe were those excavated at the Archbishop's Palace in Trondheim (Norway). They were dated to the period 1500-1537, and appear related to the minting operations carried out at the site. Over one thousand crucible fragments were recovered, representing different sizes and shapes, some of which show grainy surface textures similar to the typically Hessian wares (Saunders 2001: 27-28 and 85, fig. 5a). The crucial clue for their ascription, however, is given by the three star-like stamps found on the base of one of them, identical to the two on a crucible found in Großalmerode and illustrated by Stephan (1995: 47, fig. 39). This finding establishes the presence of Hessian crucibles in Scandinavia over two hundred years before the first written record of this trade.

Other examples of Hessian crucibles have been published by Cotter (1992) in his catalogue of triangular crucibles. Firstly, this author records two nearly identical nests of five triangular crucibles each, one found in Colchester High Street (UK), and another one from Great Yarmouth (Norfolk, UK). They are presumably unused, supporting the idea that these nests were bought as a 'unit'. In both cases, the biggest vessel is stamped in the base with the letters CG inside an oval cartouche (Cotter 1992: 267-269). Although no exact parallel for this stamp has been found, a very similar one, with the initials IG, is known from Hessian examples (Stephan 1995: 36, fig. 24). This, in addition to the characteristic thin walls and grainy texture, endorses the ascription of the English finds to the German producer.

Another crucible published by Cotter was found in London Broad Street and dated *ante quem* to 1647 (Museum of London Context No. BRO 90 [205] (353), cf. Cotter 1992: 259). Its stamp was described as not very clear, and was tentatively read as a circumference containing the letters PIV and GHK (the two last letters as a ligature). Most likely, the actual reading of the stamp is PTV GER, which would make it identical to that in contemporary crucibles found in Hesse (Stephan 1995: 26-37, figs. 24-25) and Jamestown (see above, fig. 149). Other triangular, orange, seemingly Hessian, crucibles

have been identified by the author of this thesis in the stores of the Museum of London Archaeology Service (MoLAS)[34]. Nevertheless, they generally lack clear contexts and chronologies, thus adding little to this picture. A further example of Hessian wares found in London is probably a high-fired, quartz sand-tempered late medieval crucible analysed by Freestone and Tite (Freestone and Tite 1986: 53) and whose matrix chemical composition, obtained by the same technique as used in this thesis, falls within the same range (Table 17, p. 223)[35].

Cotter (1992: 261-262) also reports some crucibles found in Koksijde (West Flanders, Belgium) and probably dated to the 17th century, which he notes as very similar to those from Colchester (see above). Accordingly, by association, these could also constitute Hessian vessels.

The inventory of possibly Hessian crucibles concludes with a group of ten unused vessels, representing six consecutive size grades, found at the mouth of the Guadalquivir River in Sanlúcar de Barrameda (Cádiz, Spain) and published by Amores Carredano and Lloret Marín (1995). Their careful description of the appearance of the crucibles – acutely patent in their characterisation of the paste surface as "toad skin texture" –, alongside the illustrations provided, and together with the context of the finds, supports a Hessian origin. These crucibles may have been swept to the seashore from a ship that would have wrecked at this difficult spot of the Atlantic route to Seville. Amores Carredano and Lloret Marín (1995: 266) note that Seville held a commercial monopoly for trade with the Indies between the 16th and the 18th centuries, and therefore even German goods would have to stop over in Seville prior to shipping to Central or South America. It is worth remembering, nevertheless, that archaeological excavations at La Isabela (Dominican Republican), the first Spanish colony in America, have not uncovered bright Hessian crucibles, but black, probably Bavarian, ones (see section 7.3.3).

[34] The accession numbers of the most obviously Hessian ones are 11437, 16752 and 17845.

[35] Another late medieval crucible analysed by Freestone and Tite (1986: 43, No. 25843) shows a very similar clay composition, but no details are given regarding the paste microstructure, therefore this sample is left outside the present catalogue.

8.3.2. Manufacture, appearance, technical standard and performance

In the light of the scientific study of a wide array of Hessian crucibles, we can attempt a synthesis of their formal and material properties, as well as an assessment of their performance.

The first noteworthy feature is the very consistent appearance of the Hessian wares, so long as they were unused. In all cases, the general appearance of the paste is orange, but a closer look reveals a grey glassy matrix with orange regions around the sand grains. These frequently erupt through the surfaces, bringing about a characteristic pimply, toad skin-like, texture. Examples of unused crucibles from Großalmerode, Porto, Cádiz, Trondheim and several English sites illustrate this. No doubt, as it happened with the black, smooth crucibles from Bavaria, the light, sandy crucibles from Hesse could have been easily identified by potential users as belonging to the same group[36].

With different conditions of use, however, this external appearance would be altered. The fabrics of used crucibles no longer show the orange shade, but normally are light grey instead. Their surfaces often appear smoother and even black in colour, as a result of the high vitrification, probably fostered by the fuel ash and metal oxide fumes, as well as deliberately reducing conditions of use for some operations. These features have been recorded in used crucibles from Cripplegate, Jamestown and Burgsteinfurt, which could not have been recognised as Hessian fabrics without scientific investigation.

Just as the appearance of the crucibles seems to have been very uniform, so does the manufacturing process. All the Hessian crucibles analysed, irrespective of their date, were produced with the same type of fabric. This was a very lean and homogeneous clay matrix, characterised by a superior alumina concentration, together with very low iron oxide, alkali and earth alkali levels. The clay base was invariably mixed with fine sand temper (20-40 vol%), well sorted (usually 200-500 μm large) and very 'clean' in that it virtually consisted of pure quartz. This description applies to practically all of the crucibles analysed, and denotes that Hessian crucible makers historically adhered to a

[36] The only potentially confusing crucibles could be those manufactured by John Dwight at Fulham, England, in the late 17th century, since he deliberately endeavoured to imitate the Hessian wares (see above, section 8.1). However, even though Dwight's crucibles admittedly look very similar to the truly Hessian ones, their spread seems to have been very limited (Green 1999: 95-98). Nowadays, the English ones can be singled out under the microscope because their fabrics contain crushed glass, added to promote the vitrification of the clay matrix (Freestone 1991).

successful recipe, using both clay and sand specifically selected for technical ceramics. Only one of the crucibles from Jamestown shows what might constitute grog temper, and this would match an alternative recipe recorded in an 18th-century source. Quartz temper aside, mineral inclusions within the crucible fabrics are generally scarce and very small. These are most often iron oxides, although nodules rich in silica, alumina and iron oxide, and potassium feldspar inclusions were also identified. Invariably, they appear dissolving within the clay matrix and fluxing the surrounding area, but never catastrophically. Only one known Hessian crucible has been reported to have nearly failed due to spot melting (Ch. Salter in Hull 2003: 11) and, at present, this may be considered an exception. Those crucibles were fired in a very strong, long and oxidising fire, reaching temperatures around 1500 °C. This firing temperature would rarely be reached during normal use of the vessels.

The material properties of the crucibles thus made would generally be advantageous. The high-alumina, low-alkali clay would render it an exceptionally refractory fabric, both thermally and chemically. Furthermore, the continuous vitrification resulting from the high temperature prefiring would make the crucibles more stable through the initial stages of firing during use, and it would increase their tensile strength and imperviousness. Even in those crucibles with heavier residues of use, there is slag mechanically penetrating through the fabric cracks, but the chemical composition of the ceramic matrix shows relatively little contamination in all cases (see Appendix 1), suggesting that the vitrification improved their resistance to chemical attack. Finally, the abundant sand temper would significantly enhance the toughness and thermal shock resistance. It was crucial that this sand was composed of quartz rather than other, less refractory, minerals, and crucible makers seem to have been aware of this. Indeed, this temper recalls the "nice white sand" recommended by Lazarus Ercker for crucible making (Sisco and Smith 1951: 24). All in all, the 'mystery' of the quality of the Hessian wares seems to have been the selection of very refractory clays, well refined and mixed with fine quartz sand, and subsequently very high fired.

In certain circumstances of use, however, the material properties of these crucibles may have been challenged. Firstly, the toughness and thermal shock resistance of the vessels may not have been sufficient in some cases, especially if they were used repeatedly: the high vitrification of the clay matrix would make it relatively prone to the development of cracks upon sudden thermal or mechanical stresses; while the quartz

inclusions, given their subspherical shape, would not arrest these lines of fracture as effectively as other, more angular, temper types (Kilikoglou *et al.* 1998; Tite *et al.* 2001).

Secondly, the grainy texture of the crucibles would present a relatively large surface area, with little cracks in the spots where the sand grains erupted (fig. 159). This would arguably facilitate the penetration of corrosive substances into the crucible body, which could also slip through the substantial shrinkage cracks along the walls, as seen above in several used vessels (figs. 146, 157, 168 and 172) (cf. Freestone and Tite 1986: 58). Some of the later beaker-shaped crucibles from Hesse, as found in Großalmerode and, quite possibly, in Oxford (for which analytical confirmation is pending), show slightly smoother surfaces. At present it is not possible to determine whether this is a result of finer temper or a more careful surface finish, but this might constitute an attempt at coping with chemical attack. In any case, when confronted to the analytical study, it seems paradoxal to recall the eulogistic statements by the chymists Johann Glauber (1651: 313) or Robert Plot (1677: 250), who emphasised that Hessian crucibles retained efficiently "metals and salts", or "the Salts and Spirits of the Chymists" (see further discussion in chapter 10).

On the contrary, limited resistance to temperature changes and vulnerability to corrosion by lead and lead oxide were precisely the two flaws of the Hessian crucibles noted by Gatterer (Gatterer 1790, as cited in Stephan 1995: 31), but he offered some advice so as to minimise them: to prevent unexpected fractures, he suggested using soft crucible bases, increasing the temperature of the furnace gradually, and placing the crucibles back in the furnace after casting for a more steady cooling; to counter corrosion, he proposed smearing the crucible with oil and sprinkling some crushed green glass. This would have provided a surface vitrification layer probably reducing the open porosity of the surfaces. Obviously, Gatterer was unable to explain these flaws in the material properties of the crucibles using the same terms as this thesis, but he observed the problems and was acquainted with the strategies to prevent them. For present-day archaeometrists, the obvious moral of this story is the need of distinguishing material properties from performance characteristics and specific techniques of use when studying past artefacts (see section 3.6).

8.4. Summary

Since the 12th century at least, the region of Hesse has witnessed the development of a large-scale crucible manufacturing industry, with the main centres in (Groß-)Almerode and Epterode. Combining historical and archaeological information, it is possible to establish that by the early 16th century, and possibly earlier, Hessian crucibles were exported to a wide international market. Not only do we find evidence of this trade throughout Germany, but also in Belgium, Britain, Scandinavia, the Iberian Peninsula and even in colonial North America.

These vessels were used for all sorts of pyrotechnological activities, including goldsmithing, coin minting, ore assaying and bronze and brass metallurgy. This success may be related to the outstanding performance of the crucibles, praised as unique in several written sources, which even gave rise to imitations. The secret behind this quality was a careful selection of raw materials and a systematic manufacturing process that remained unchanged for centuries. Written descriptions of the crucible making process as recorded in the late 18th century match nearly word for word what archaeometric analyses document in samples predating these sources by almost two hundred years. Normally, specific clays of remarkable chemical and thermal refractoriness were collected and refined, and subsequently tempered with very fine quartz sand from the Fahren Creek. Also grog could be used as temper. After forming on the wheel and drying, the crucibles were fired in an oxidising atmosphere to extremely high temperatures.

Contrary to traditional assumptions, Hessian crucibles may or may not be triangular. What most of them share is a particularly pimply texture and a bright orange surface colour. The clay matrices of these vessels are strikingly uniform in their chemical compositions and in their appearance under the microscope, showing a highly vitrified grey matrix with orange regions around the sand grains. These features are only present in unused vessels.

Given their specific microstructure, the generally advantageous material properties of the Hessian wares may have been challenged in situations of extreme thermal shock or chemical attack. However, historical sources explain how to counter these possible flaws, thereby ensuring a good performance.

9. 'Neither dark nor bright': other crucible productions

"Not all treasure is silver and gold, mate."
Jack Sparrow (Johnny Depp) in *Pirates of the Caribbean* (2003)

9.1. Introduction

The previous two chapters addressed the history and technology of crucible production in what seem to have been the most important areas where technical ceramics were manufactured in the Renaissance and later times. It has to be emphasised, however, that other productions co-existed with the famous 'bright' crucibles from Hesse and the 'dark' crucibles from Bavaria and surrounding regions. Much more research will be needed before a precise assessment can be made of the extent to which these major industries dominated the market at different times and areas; but the presence of other, even if less important, crucible producers must be brought into the picture in order to avoid undue simplifications.

This chapter briefly summarises historical references to suitable clay banks or crucible manufacturers outside the main production centres discussed above. Subsequently, this information is completed with details of some post-medieval crucibles whose analytical characterisation led to their ascription to this broad category of 'the others'. After this, we will have enough data for a more comprehensive and interpretative approach to the production and consumption of crucibles, which will be presented in the next chapter.

9.2. Alternative crucible productions in the written sources

A survey for sources of crucibles cited in written sources reveals an array of options. It is often difficult to discern when the places mentioned are significant production centres or simply those familiar to the author in question. Furthermore, some sources point to specific clay banks whilst others directly note the provenance of

the vessels, and these – *i.e.* clay and manufacturer origins – did not necessarily coincide, as illustrated by the case of the Ipsian crucibles (section 7.2). A large number of the places cited are within the borders of present-day Germany. Although this region undoubtedly housed the largest concentration of crucible producers in the post-medieval world, this emphasis may also be a reflection of the country best known to most of these writers. Finally, along with these smaller productions, one should not forget that some users could make their own vessels, a practice recorded in several sources as late as the 17th century (Glauber 1651: 313-327, and section 10.2).

In the early 16th century, the anonymous author of the *Probierbüchlein* recommends that crucibles "should be made of blue or gray clay, or of Oberg earth from near the monastery of Hildesheim", in Lower Saxony (Sisco and Smith 1949: 111). A few decades later, the same location appears in Agricola's *De natura fossilium* as a source of "black chalk" used for crucible making. However, Agricola adds some more localities and, quite interestingly, he insinuates a functional specialisation: not only does he mention the triangular crucibles from Ips, used at the mint, but also those from Waldenburg, which are described as being very fire resistant and allegedly preferred by apothecaries, or those from Rothenburg, near Nurenberg, used for brass production. Other sources of clay for technical ceramics mentioned by Agricola are in Lower Lorraine, Aachen, Sieburg and, further to the West, in Spain (Fraustadt and Prescher 1958: 52-61). When addressing brass making, Biringuccio notes that "the crucibles were made of Valencia [SE Spain] clay, or they were brought ready-made from Vienna"; later, in his discussion of glass working, he again cites Valencia clay, but also acknowledges the use of "that of Treguanda [Tuscany, Italy] or other places whose clay resists the fire for a long time because of its particular nature" (Smith and Gnudi 1990: 72 and 128).

In the 17th century, Gabriel Plattes (1639: 19) mentions "*Flanders* melting Pots" being sold in London, co-existing with local *ad hoc* manufactures. The Flanders crucibles may have been truly Dutch vessels or, alternatively, German or other productions shipped from the Low Countries. Furthermore, even if made in Flanders, these vessels could be manufactured with imported clay, as suggested by Glauber when citing "earth brought out of *England*, and *France* into Holland" for crucible making (Glauber 1651: 313). Apart from Dutch, Hessian and "*Gibsensian*" (Ipsian?) crucibles, Glauber also credits the reputation of the crucibles from Waldenburg. This must have constituted a long lived production centre, as it was referred to by Agricola one century before (see above), and it will be cited again over a hundred year later (see below).

The 18th century witnesses further diversification in the market. In their *Encyclopédie*, Diderot and D'Alembert (as cited in Cotter 1992: 265) acknowledge that the Hessian crucibles are the most famous, but they note that French crucibles now perform equally well. In England, the production of crucibles from British materials is institutionally encouraged, and several corporations begin to manufacture them with varying success (Chaldecott 1981; Cotter 1992: 267-269). Within Germany, Gatterer (1790, as cited in Stephan 1995: 31) explains how the existing crucible makers from Hesse, Bavaria and Bohemia find growing competitors in the following areas: Charlottenburg, near Berlin; Waldenburg in Saxony; Skele, Wurgel and other places near Magdeburg; Engelshartszell im Lande ob der Ens, near the Danube; and Lutterberg, near Hanover. According to him, the productions from Lutterberg and Waldenburg were deliberate – yet unsuccessful – attempts at imitating Hessian crucibles.

The Great Exhibition that took place in London in 1851 included a competition of crucibles from across the world. The broad range of international high-quality technical ceramics presented there indicates that the 'secrets' of crucible manufacture were widely known by then (Percy 1875: 110-141). This market expansion would have been supported by the increasing demand, together with the developments in manufacturing and transportation systems in the wake of the Industrial Revolution.

9.3. Material properties and performance of some alternative crucibles

It is quite remarkable that the vast majority of the crucibles analysed within this PhD project were found to be either Hessian or Bavarian, especially if one takes into account that the selection of samples was not guided by their assumed provenance. This hints at a considerable dominance of these major productions at the expense of smaller or local producers, although much more archaeological data will be needed in order to quantify these interactions and study their evolution over time. In any case, the characterisation of the few technical ceramics found to originate elsewhere may offer some clues to explain the better reputation of the more successful manufacturers.

9.3.1. Zwickau (Saxony)

The metallurgical remains from Zwickau were dated to the second half of the 15th century (Beutmann *et al.* 2000). Two crucible types were identified: on the one hand,

big (≤400 mm high) oblong pots, used in combination with domed lids for the production of brass by cementation of metallic copper with zinc ore; on the other hand, smaller (~13 mm high) triangular vessels used for the melting and refining of brass prior to casting (Martinón-Torres and Rehren 2002) (fig. 174).

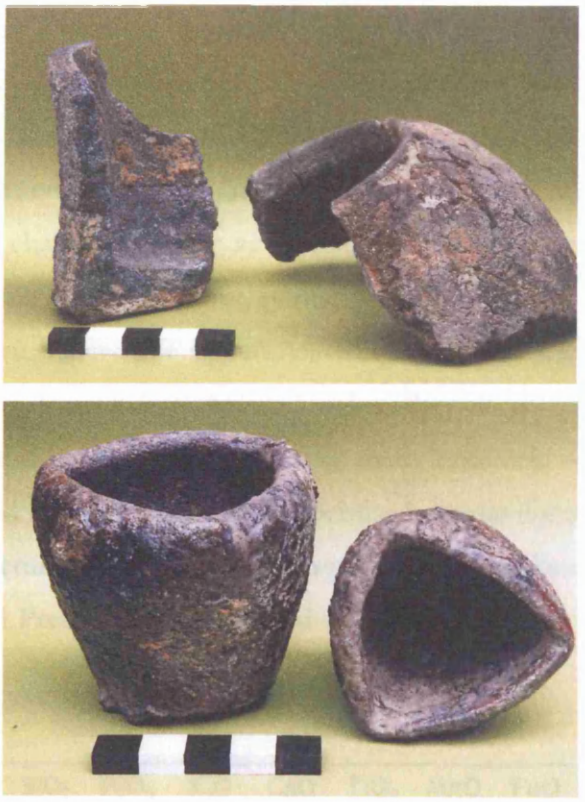


Figure 174. Crucibles from Zwickau. Top, fragments of brass cementation pots. Bottom, triangular vessels used for melting and refining. Scalebar is 10 cm (photos Jens Beutmann).

The cementation crucibles are thick (~25 mm) and appear heavily vitrified as a result of the relatively low alumina content in the clay matrix and the considerable amounts of zinc oxide contamination, which promoted the softening of the ceramic during use. The paste is tempered with ~40 vol% subangular quartz grains. The lids and the triangular crucibles are much thinner, as they would not be expected to hold the same weights. Both of them were made with a different ceramic material, being much richer in alumina, and also containing abundant quartz inclusions (fig. 175; Table 18).

From a technical viewpoint, the most special aspect of all the crucible samples is that they invariably appear coated by a layer of less refractory clay, now heavily vitrified (fig. 175; Table 18, note the generally lower alumina and higher potash content in the ceramic matrix). This was used not only to seal the lids to the bigger crucibles, but generally to provide an external 'wrap' for all the ceramics. Quite possibly, this outer layer was indeed expected to vitrify during the high-temperature utilisation, either to provide a highly viscous wrapping to counteract the effect of any cracks in the main ceramic body, and/or to distribute the heat from the furnace more evenly and thus avoid cracks in the first place (Bayley and Barclay 1990; Bayley *et al.* 1991; Martinón-Torres and Rehren 2002). This continuous outer ceramic layer is a feature frequently documented in Roman and medieval crucibles, but it has not been recorded in any of the other post-medieval crucibles analysed in this work. This may be related to the more refractory nature of the latter vessels, which would make this protective layer

unnecessary. For earlier and less refractory crucibles, this procedure would ensure an acceptable performance in spite of the limited material properties of the ceramic.

As it seems, these crucibles could have been produced locally. The remarkably high titania content in the crucible matrices could be interpreted as a geological signature of the hinterland of Zwickau, characterised by an abundance of titanium minerals (Martinón-Torres and Rehren 2002: 108). Moreover, perhaps the metallurgists themselves made their own crucibles, if we believe the recommendation made by the author of the *Probierbüchlein*: "If you want to learn it [crucible making] really well, go to the bronze founders or those who cast and work brass; they make many crucibles – though not all of them well." These would co-exist with more specialised productions such as the brass making crucibles manufactured in Rothenburg – not far from Zwickau – and reported by Agricola (Fraustadt and Prescher 1958: 53) and Libavius (Rex 1964: 12).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
Zwickau big	-	0.8	17.9	67.0	0.4	2.1	0.9	2.9	0.1	7.9
Zwickau lid	-	0.4	24.4	66.1	0.4	1.5	0.3	4.0	<0.1	2.8
Zwickau triangular	-	0.4	24.1	64.4	0.4	1.8	0.5	4.1	<0.1	4.3
Wrap big	-	1.6	16.9	63.7	0.8	6.0	3.4	3.8	0.4	3.3
Wrap lid	-	0.8	23.5	66.0	0.5	4.4	0.3	1.5	0.1	3.0
Wrap triangular A	-	1.2	16.1	68.0	0.7	6.1	2.0	1.6	0.3	4.0
Wrap triangular B	-	0.5	16.6	70.0	0.4	3.3	0.8	6.4	0.1	2.0
Oberstockstall mean	0.2	0.7	32.4	57.3	0.3	2.1	0.7	1.3	-	5.0
Obernzell OBZL 01	1.1	1.0	27.7	58.3	0.4	2.5	0.9	1.2	-	6.9
Oxford OX 1422	0.3	0.8	28.9	55.5	0.2	2.6	0.9	1.4	-	9.3
Hesse mean	0.1	0.5	36.9	56.8	0.2	1.3	0.3	1.9	-	2.0

Table 18. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of the bodies and outer 'wraps' of the crucibles from Zwickau. In the bottom rows, the composition of other crucibles discussed in this thesis is given for comparison (data for Zwickau samples from Martinón-Torres and Rehren 2002; note that Na_2O was not measured due to the line overlap between the Zn Lα and the Na Kα lines, and that the figures given were re-normalised after neglecting contamination by ZnO, CuO and PbO).

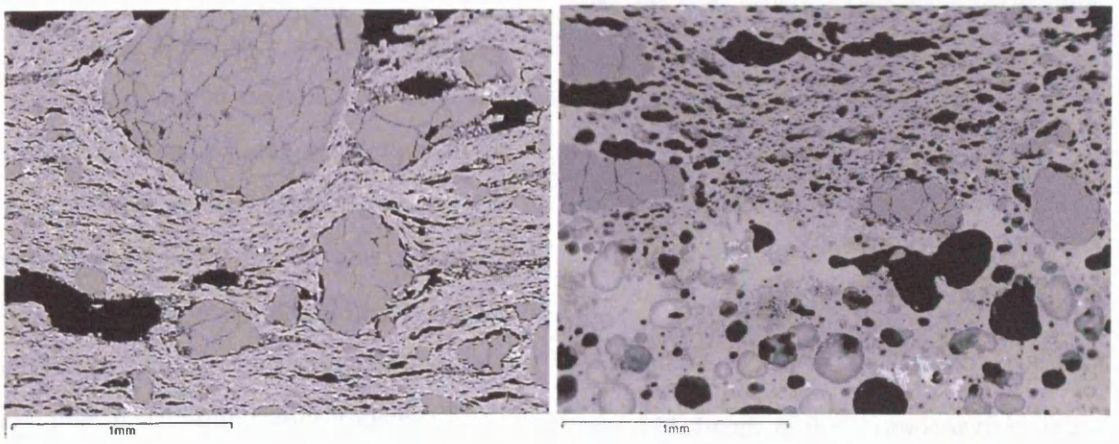


Figure 175. Left, fabric of one of the crucible lids from Zwickau, tempered with quartz grains (Z-29/319/s2, BSE, 50x). Right, interface between the body of a triangular crucible (top), and the outer, much more vitrified, ceramic layer (Z-29/317/s1, BSE, 50x).

9.3.2. Cripplegate Buildings (London, United Kingdom)

The technical remains from Cripplegate contain crucibles that could not be ascribed to either of the major 'bright' or 'dark' categories defined in the previous chapters, together with other vessels that were positively identified as 'bright Hessian' (see section 8.3.1).

Figure 176. Inner surface of the triangular crucible WFG 18/114 from Cripplegate. Note the smooth finish and the marks from turning on the wheel.

One of them (WFG 18/114) is a triangular crucible, buff-coloured, with a smooth finish but showing clear horizontal striations from its manufacture on the wheel. The rim is ~70 mm wide, and the wall has a thickness of ~4 mm (fig. 176). XRF analyses showed low signals of zinc, copper and lead (Bayley 2003: 2), but this contamination was not detected by SEM-EDS. The vessel has no visible traces of high-temperature reactions. The paste shows no vitrification, and the layered texture of the clay minerals is clearly noticeable (fig. 178). It contains a variety of subangular to spherical mineral inclusions, including quartz, hornblende and other silicates, as well as zircon, ilmenite and rutile (fig. 177). Compositionally, the ceramic matrix exhibits a high alumina concentration, combined with low alkali, earth alkali and iron oxide levels

(Table 19). In this sense, it may be considered a suitable raw material for crucibles, being of sufficient thermal and chemical refractoriness. However, owing to the low temperature prefiring, the vessel may have lacked the necessary tensile strength for holding heavy metals, while the lack of vitrification would have resulted in a limited imperviousness.

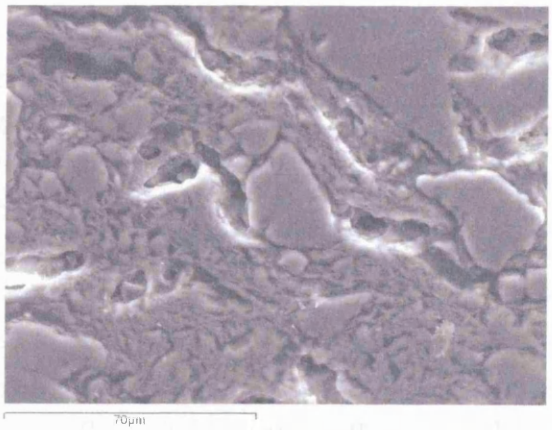


Figure 178. Image of the ceramic matrix under high magnification, showing the lack of vitrification indicating a relatively low temperature prefiring (WFG 18/114/s1, SE, 800x).

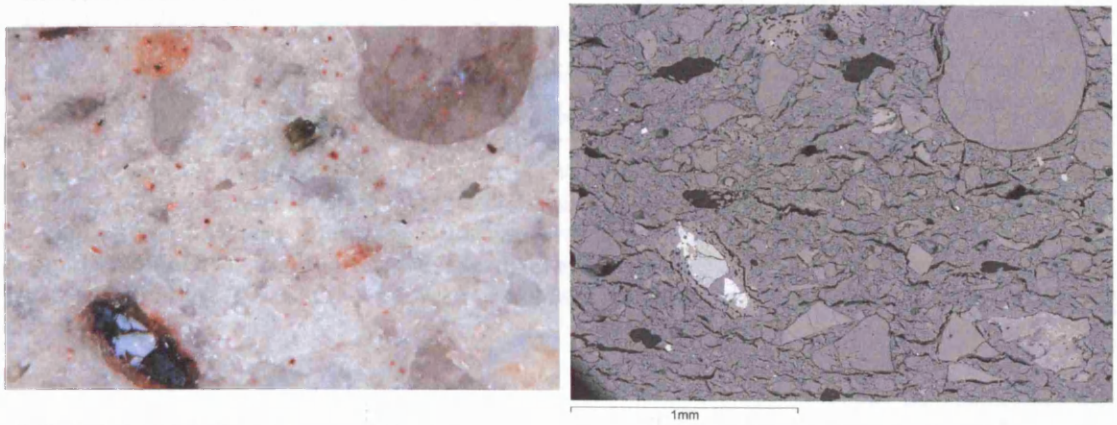


Figure 177. Two views of the same area of the ceramic fabric of the crucible, showing the variety of mineral inclusions and the low vitrification of the ceramic matrix (WFG 18/114/s1, left XPL, 50x, long axis represents ~2 mm; right, BSE, 50x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
Zwickau big	-	0.8	17.9	67.0	0.4	2.1	0.9	2.9	0.1	7.9
Zwickau lid	-	0.4	24.4	66.1	0.4	1.5	0.3	4.0	<0.1	2.8
Zwickau triangular	-	0.4	24.1	64.4	0.4	1.8	0.5	4.1	<0.1	4.3
WFG 118/114	0.8	0.9	24.4	67.7	0.4	2.1	0.7	1.4	-	1.7
WFG 118/107	0.2	0.2	17.9	77.8	0.1	1.2	0.4	1.2	-	1.0
WFG 118/121	0.3	1.2	22.6	66.1	0.1	4.3	0.5	1.0	-	3.8
London 25836	-	1.2	26.4	59.6	0.8	3.4	2.3	0.8	-	4.5
London 25837	0.8	0.6	28.4	61.2	0.9	1.7	2.0	1.0	-	3.3
London 25838	-	1.1	24.8	64.9	-	3.0	0.6	1.1	-	3.9
London 25839	-	1.4	22.2	66.0	-	2.5	1.2	0.7	-	4.6
London 25840	-	1.2	25.2	61.7	0.6	2.5	1.4	0.7	-	5.5
London 25841	-	0.9	25.3	66.7	-	2.5	1.3	0.8	-	2.1
London 25849	-	1.1	26.0	65.6	-	3.2	0.4	0.9	-	2.4
Weyerstraße D-94/4	0.5	0.5	25.3	65.6	0.5	3.0	0.8	1.7	-	2.1
Oberstockstall mean	0.2	0.7	32.4	57.3	0.3	2.1	0.7	1.3	-	5.0
Obernzell OBZL 01	1.1	1.0	27.7	58.3	0.4	2.5	0.9	1.2	-	6.9
Oxford OX 1422	0.3	0.8	28.9	55.5	0.2	2.6	0.9	1.4	-	9.3
Hesse mean	0.1	0.5	36.9	56.8	0.2	1.3	0.3	1.9	-	2.0

Table 19. Average chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of the different crucible samples discussed in this chapter. The composition of medieval crucibles from London, and that of other crucibles discussed in this thesis, is given for comparison (data for London crucibles from Freestone and Tite 1986: 42). Note that the figures given for WFG 118/107 and D-94/4 were re-normalised omitting, respectively, 0.7 wt% ZnO and 0.5 % Cl.

Figure 179. Sample WFG 18/121 from Cripplegate.

The next ceramic sample from Cripplegate was removed from a vessel that does not show any evidence of high-temperature use or metal contamination. Unlike the triangular vessel discussed above, its shape is not diagnostic enough as to ascertain whether it actually constitutes a crucible. It is a small, wheel-thrown, subcylindrical vessel, about 40 mm in diameter and slightly opening towards the top (fig. 179). The considerable thickness of the body (~7 mm), however, suggests that it may constitute a technical ware. The paste shows a buff colour that becomes bright

brown on the outer surface as a result of an oxidising firing. Two features of the fabric are the most prominent: on the one hand, its fine-grained texture; on the other hand, the complete lack of vitrification (fig. 180). Silicate inclusions are extremely abundant (50-60 vol%), but their diameter rarely exceeds 100 μm. These mostly consist of quartz, together with a few feldspars. Minuscule ilmenite and rutile particles are also present. The chemical composition of the clay matrix is comparable to that of the previous crucible (Table 19), contrasting to the different nature and abundance of the mineral inclusions. In its composition and microstructure, this fabric appears analogous to some medieval crucibles made in London (Freestone and Tite 1986; Bayley *et al.* 1991), therefore it may reasonably be assumed that it constitutes a local production as well (Table 19).

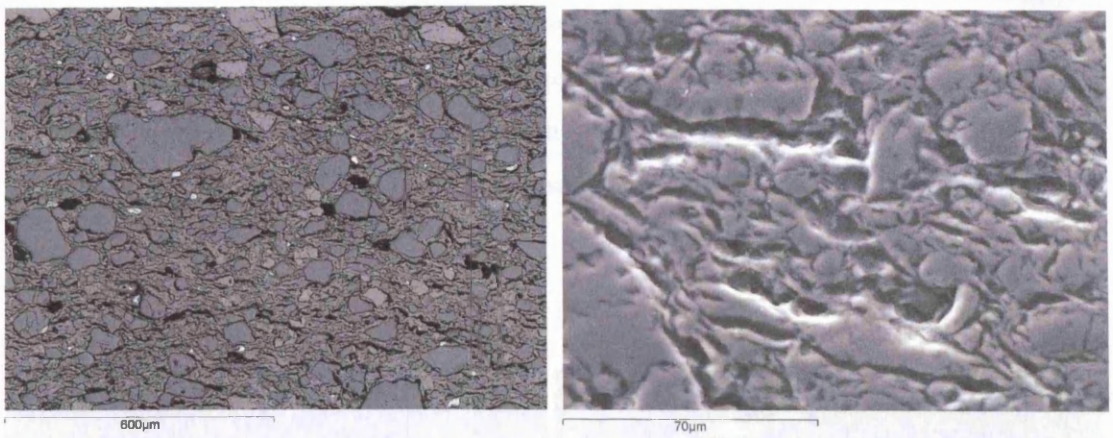


Figure 180. Two views of the ceramic fabric of the vessel. Note the fine-grained texture and the outstanding abundance of small quartz inclusions, as well as the lack of matrix vitrification (WFG 18/121/s1; left, BSE, 50x; right, SE, 800x).

The last specimen is one of two sherds resembling stoneware fabrics (WFG 18/107), slightly curved but too fragmentary to allow a reconstruction of the original artefact's shape (fig. 181). The outer surface is covered by a bright, black glassy layer, clearly secondary, which prevents an estimate of the original surface finish. The paste is grey and very fine-grained, although some big inclusions are present. It contains ~30 vol% silt-sized quartz grains, and innumerable minuscule zircon and rutile particles. Quartz inclusions appear buckling as a result of the high temperatures, and the smallest ones are dissolving in the fabric. Partly as a result of this, the composition of the glassy phase shows comparatively high silica concentrations, even though bigger quartz grains were avoided during analyses. The ceramic matrix shows continuous vitrification, with fine to medium voids (fig. 182). A grainy, vitrified, purple sediment covers the inner

surface, although there is very little interaction between this and the fabric. Microscopic and SEM-EDS examination shows that this layer is enriched in lime, potash and soda, while it contains abundant concentrations of copper oxide (~95 wt% in some phases), minor amounts of zinc oxide (≤3 wt%), and discrete quartz inclusions (fig. 183).

The analogies between this specimen and some stoneware fabrics analysed by Freestone and Tite (1997) suggest that this may constitute a fragment of a stoneware vessel that was re-used for metallurgical purposes, taking advantage of the well fired, thick and sturdy fabric. The composition of the inner layer may constitute a mixture of an existing alkali glaze with metallic residues, although this cannot be confirmed.

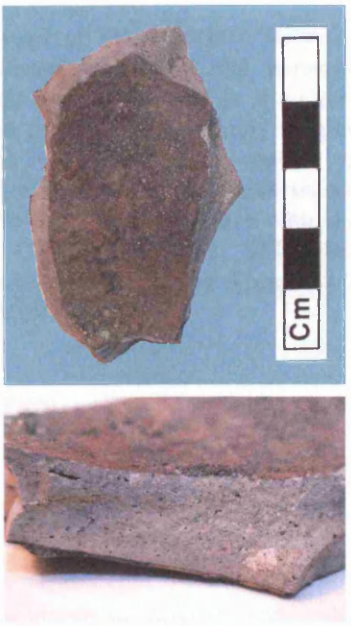


Figure 181. Inner surface (top) and section (bottom) of the sample WFG 18/107. Note the presence of a dense, vitrified layer adhering to the inner surface.

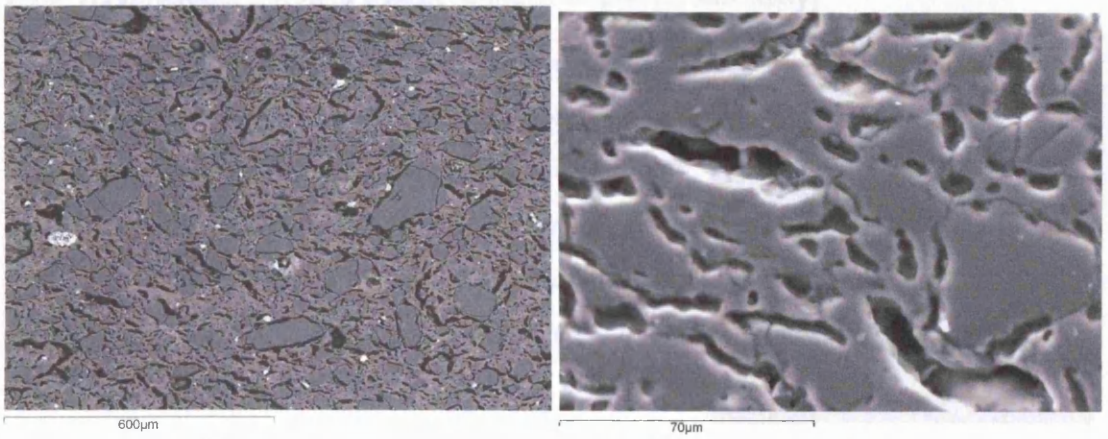


Figure 182. Ceramic fabric of the stoneware ceramic used for metallurgical purposes. Left, note the very small size of the quartz inclusions, in spite of the relatively high magnification of the image. Right, detail of the vitrification of the ceramic matrix, showing fine to medium voids and the melting of quartz grains (WFG 118/107/s2; left, BSE, 100x; right, SE, 800x).

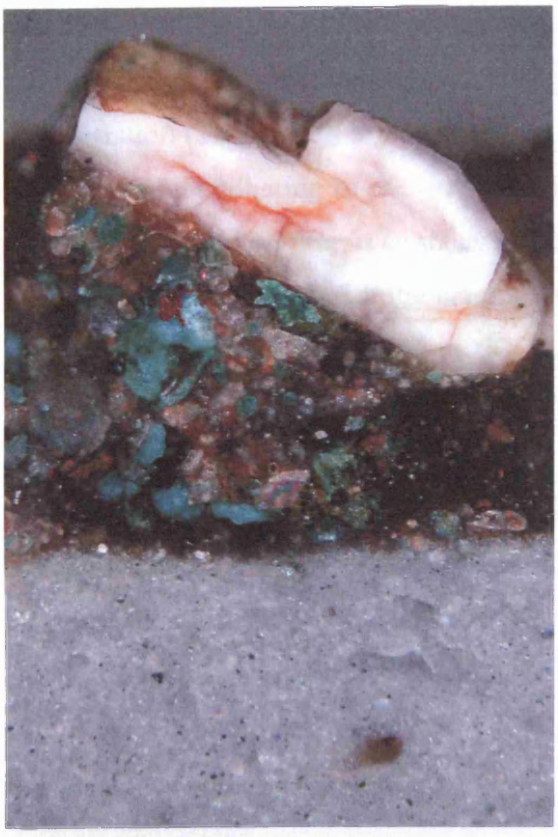


Figure 183. Detail of the interface between the inner sediment (top) and the ceramic body. The large white particle is a quartz grain, the green phases are primarily copper carbonate, and the translucent matrix is richer in alkali elements and contains traces of zinc oxide. Note the neat interface with the ceramic fabric (WFG 118/107/s2, XPL, 50x, long axis represents ~2 mm). See Appendix 2 for analytical details.

9.3.3. Weyerstraße, Cologne (North Rhine-Westphalia, Germany)

Previous analytical work on the remains of the Renaissance goldsmith's workshop from Weyerstraße had concentrated on the residues of use (Rehren 1996a; Rehren 1996b). For the present work, the only sample of technical ceramic that could be obtained was a mounted specimen comprising a section through a rim with adhering lute. Given its incompleteness, an estimate of the original shape of the artefact is not feasible (fig. 184).

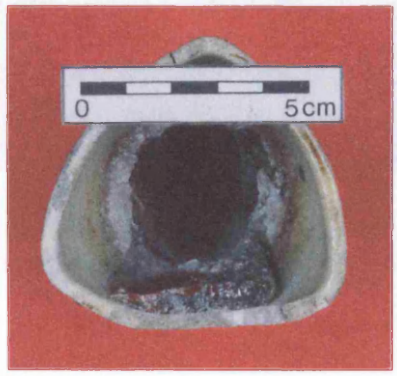


Figure 184. Top view of a triangular crucible from Weyerstraße. Note that the specimen discussed in the text is not necessarily from a vessel like this (photo Thilo Rehren).

The fabric of D-94/4 consists of a grey, fine-grained, very compact matrix, containing abundant (~30 wt%) and generally large (0.5-1 mm) mineral inclusions. These are mostly quartz grains, but also feldspar and iron oxide minerals are present, together with smaller rutile and zircon. The paste shows shrinkage cracks across the body and only initial vitrification, with no melting minerals. This indicates a

relatively low temperature for firing and use (fig. 185). Its chemical composition resembles that of the London crucibles, although with a slightly higher titania concentration (Table 19).

The lute adhering to the outer surface and the tip of the rim presents a feature that is not easy to interpret: it contains several prills of pure metallic lead. Clearly, as this metal oxidised, it reacted with the ceramic matrix to form a lead silicate glass. There is a surprisingly clean interface between this glassy lute and the main body of the vessel, suggesting that the lead formed part of the lute before it was smeared on the main artefact, perhaps in a deliberate attempt to foster its vitrification (fig. 186). A plausibly similar practice may be that recorded by Agricola (1558: 133-134) when describing brass making techniques, and observing the mixing of *ferri squama* [hammer scale?, potentially a strong flux for silica-rich materials] with the clay to be used as external lute (cf. Martinón-Torres and Rehren 2002).

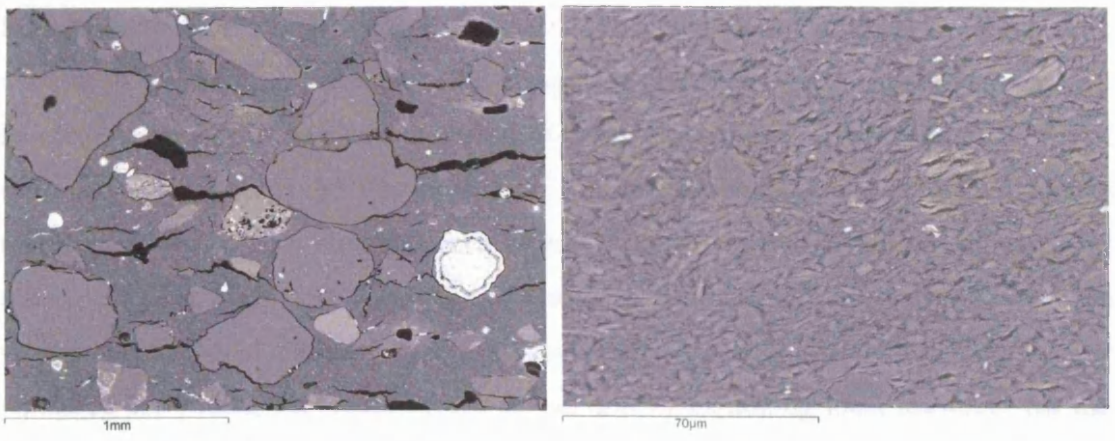


Figure 185. Ceramic fabric of the technical ceramic. Left, note the abundance and large size of the inclusions. Right, detail of the ceramic matrix showing very limited vitrification (D-94/4/s1; left, BSE, 50x; right, SE, 800x)

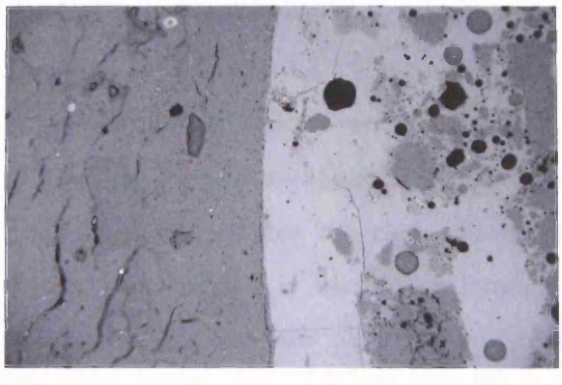


Figure 186. Detail of the interface between the main ceramic body (left) and the outer lute layer. Note the lack of chemical interaction between the two, and the glassy condition of the lute, mostly consisting of lead silicate (D-94/4/s1, PL, 50x, long axis represents ~2 mm).

9.4. Summary

Even though the market of technical ceramics in the Renaissance was dominated by the bright crucibles from Hesse and the dark crucibles from Bavaria and surrounding regions, these productions co-existed with vessels manufactured at a much smaller scale, possibly locally and, in some cases, by the users themselves. Alternative crucibles were made even in regions near the most famous German producers.

Different authors refer to a variety of production centres or suitable clay deposits familiar to them, and written information also reflects that the small-scale manufacture of technical ceramics did not decline over time, in spite of the expansion of the more established industries. Interestingly, some sources imply the specialisation of some centres in the production of crucibles for specific pyrotechnological activities such as brass making or coin minting.

The archaeological and analytical study illustrates locally produced vessels whose design was adapted to their specific utilisation, as was the case for brass making and brass melting crucibles. In general terms, it seems that these technical ceramics were made of a considerably refractory clay, tempered with sand and fired to comparatively low temperatures, probably not exceeding 850 °C, *i.e.* the range of firing temperatures of most domestic pottery at this time. The chemical composition of some ceramic matrices, however, with alumina levels below 20 wt%, indicated a more limited heat resistance. It is likely that these crucible makers resorted to raw materials available nearby, which would generally present sufficient material properties. In this sense, they would be continuing the earlier medieval tradition of exploiting local resources for the production of metallurgical ceramics. Even if the technical quality of these crucibles may be seen as generally acceptable – provided that they were not subjected to extreme conditions of use –, the clays employed were probably within the same range as used for producing ordinary pottery, as has been shown for the case of medieval crucibles from England (Freestone and Tite 1986; Freestone 1989; Bayley *et al.* 1991), whose composition resembles closely that of some vessels studied here.

In Cripplegate, Hessian wares were used alongside alternative, possibly local, crucibles, and possibly recycled ceramic containers such as stoneware vessels. The resulting impression is that of craftsmen maximising the available resources, maybe opting for different raw materials and tools at different times. The potential reasons behind those choices will be addressed in the next chapter.

10. The production and consumption of crucibles in the post-medieval world: a global perspective

"The trouble with facts is that there are so many of them."
Samuel McChord Crothers (1857-1927)

"If you choose not to decide – you still have made a choice!"
Neil Peart (1952-1997)

"The individual choice of garnishment of a burger can be an important point to the consumer in this day when individualism is an increasingly important thing to people."
Donald N. Smith, president of Burger King (2003)

10.1. Introduction

The information collected up to this point has allowed the identification of two main manufacturers of technical ceramics in Renaissance and later Europe, whose products were traded across the continent and beyond (cf. Martinón-Torres and Rehren 2004c) (fig. 187). Dark and bright crucibles were different in their origins and design, but they served similar purposes with generally equivalent technical performance. They probably competed in the market with each other, as well as with smaller-scale productions. So far, the focus of the thesis has been the characterisation of the formal and material properties, alongside the performance characteristics, of the various crucible types. In each case, additional observations have been made regarding the manufacturing process, the choices made by producers and consumers, the geographic and operative span of the various wares, and the perception of raw materials and end products by the different agents involved.

Now it is time for a more comprehensive view. This chapter presents a broader picture of the production and consumption of crucibles, where a larger number of variables are considered comparatively for the different types of vessels. In the first section, the process of crucible manufacture will be addressed in a sequential fashion, as a *chaîne opératoire* that starts with the selection of raw materials and reaches a crucial point when the product is ready to be sold. Different ways of making crucibles will be discussed by systematically contrasting written, archaeological and analytical

information. In the second section, an attempt will be made to explore the reasons that might lead to the choice of a particular crucible type in a given context. Notwithstanding the structure of this chapter, production and consumption will necessarily be seen as two sides of an adaptive and recursive negotiation taking place in specific social and cultural arenas (cf. Cumberpatch 1997b; Sillar 2000b: 8-13). Finally, on the basis of this complex historical, social and technological background, an attempt will be made at determining why the most renowned crucible production centres achieved their success. From an epistemological perspective, the role of perception and nonverbal cognition in the study of human choice will become apparent.

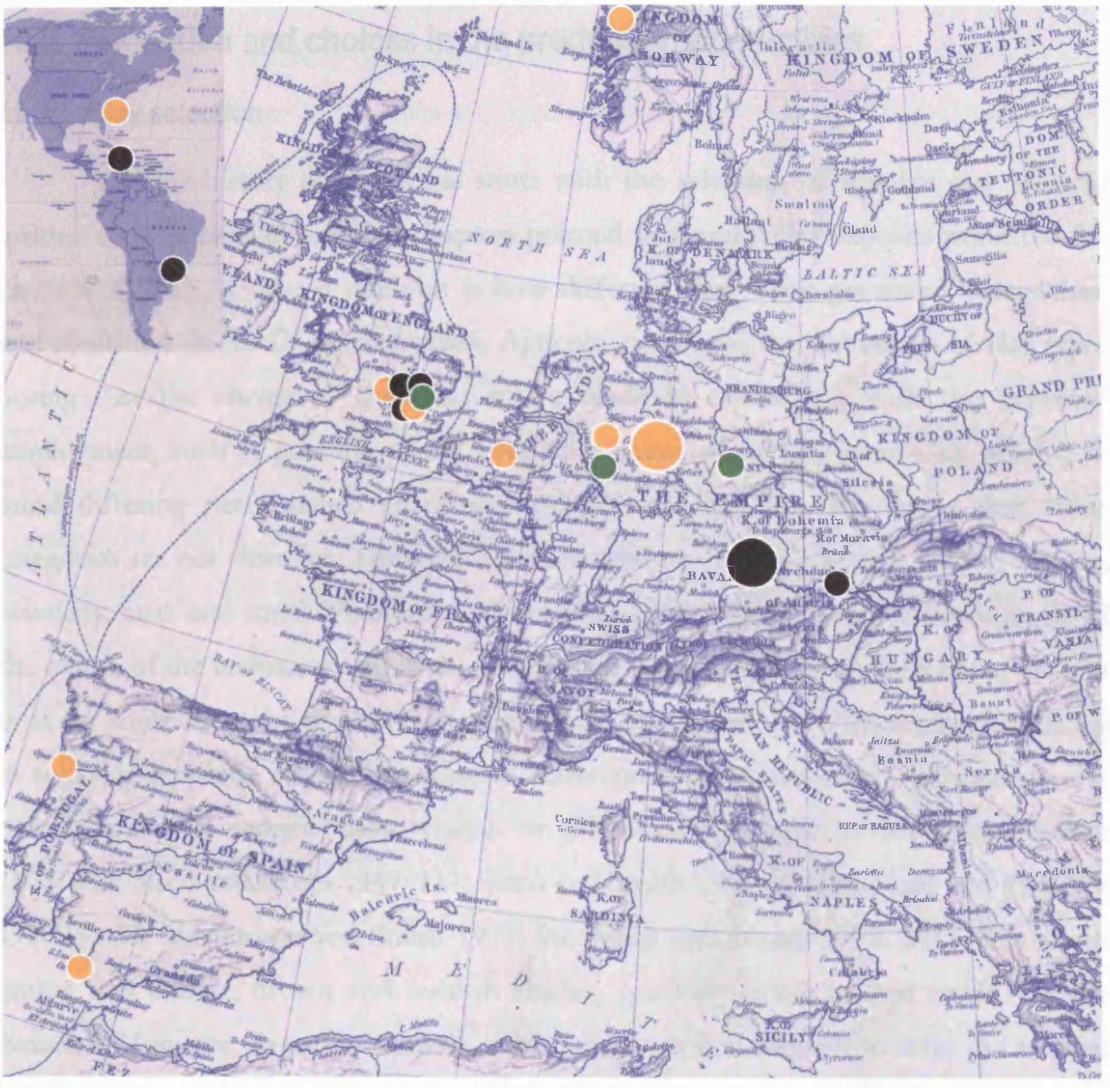


Figure 187. Geographic distribution of the archaeological crucibles discussed in this thesis, with an indication of their attribution. Orange circles denote Hessian, black ones are Bavarian, and green ones are 'alternative productions'. Bigger circles are production centres. Note that the circle for Trondheim (Norway) should be further to the North, outside the area covered by this map (after Shepherd 1923, modified).

An important dimension to be considered in a wider perspective of the production and consumption of crucibles is the institutional and geographic channels of transport and trade of these commodities across post-medieval Europe and beyond. This may be informative of the relations amongst different spheres of technology and the broader social and economic implications of this trade, while it also contributes to explain the international success of some manufactures. However, such a complex topic deserves dedicated research on its own merit and cannot be addressed within the constraints of this PhD project.

10.2. Perception and choices in the production of crucibles

10.2.1. Clay selection

The life-history of a crucible starts with the selection of suitable clay. Several written sources cited in previous chapters pointed to specific clay deposits preferred for crucible making. A related question is how different clays were perceived, categorised and identified. In his *De natura fossilium*, Agricola introduces the discussion of clay types noting that the choice of clays has to be made in accordance with the expected employment, such as growing plants, preparing paints or making pots – *i.e.* bearing in mind differing performance characteristics. Nonetheless, he also warns that these categories are not clear-cut. The main features used to discern clays are colour, texture, plasticity, taste and smell (Fraustadt and Prescher 1958: 44-45). No mention is made of the nature of the inclusions within the clay, which supports the idea raised above that, at least for some time, the presence of graphite flakes may not have been a leading factor in selecting graphitic clays. According to different authors, seemingly appropriate clay colours are those ranging from whitish or yellow through grey to blue (e.g. Glauber 1651: 314; Sisco and Smith 1949: 111; Sisco and Smith 1951: 24; Fraustadt and Prescher 1958: 52-53; Hawthorne and Smith 1979: 96; Smith and Gnudi 1990: 391). It is worth noting that orange, brown and reddish shades, generally richer in iron oxide, are not included. However, from the analysis of end products it is difficult to infer the original colour of the clay used, therefore the study of archaeological crucibles has little to add to this point.

Along with the consideration of *how* clays were singled out, another pertinent question is *why* they were selected. A very important requirement seems to have been

thermal stability, as acknowledged by several authors (e.g. Sisco and Smith 1951: 180; Fraustadt and Prescher 1958: 52; Smith and Gnudi 1990: 128). Thomas Norton (1652: 95) puts it aptly: "But manie Claies woll leape in Fier, / Such for Vessells doe not desire". This concern with the thermal refractoriness of the crucibles, widely conspicuous in written sources, somewhat contrasts with archaeological studies suggesting that the availability of clays resistant to high temperatures may not have been such a big constraint, given that the majority of the crucibles used since Roman times appear stable up to 1200 °C (Freestone and Tite 1986; Bayley *et al.* 1991; Jackson *et al.* 2003). The analyses carried out for this thesis show that the most successful crucibles were invariably very rich in alumina – hence very refractory –, but the alternative productions were generally heat resistant as well, even if not as much. This apparent paradox may be related to the consumption, use and performance of the vessels, and it will be discussed later in this chapter.

10.2.2. Paste preparation

Having collected the clay, the next step would be preparing the paste. This might involve a refining stage as well as the mixing of the clay matrix with temper. Biringuccio advises that the clay "be well freed from small stones and beaten well" (Smith and Gnudi 1990: 391). It was shown above how the crucibles from Oberstockstall indicated that the raw material was probably levigated so that coarser fabrics were reserved for increasingly larger crucibles. Also the clay used for Hessian crucibles was routinely refined.

Two tempering materials appear recurrently in the textual sources: on the one hand, sand or crushed quartz; on the other hand, grog. From the 12th century at least, different authors mention either of the two options, or both of them (e.g. grog in Glauber 1651: 315; Norton 1652: 95; Sisco and Smith 1949: 111; Hoover and Hoover 1950: 230; Wyckoff 1967: 51 and 230; Hawthorne and Smith 1979: 142; and sand in Sisco and Smith 1949: 111; Sisco and Smith 1951: 24). Both practices are documented archaeologically, with grog present in some of the scorifiers and other wares from Oberstockstall, as well as in one of the Hessian crucibles found in Jamestown, and quartz sand prevailing in most of the other technical ceramics. The co-existence of both recipes has also been recorded in late medieval crucibles found in Britain (Freestone and Tite 1986), Germany and France (Martinón-Torres 2001). Freestone and Tite (1986: 58)

noticed a general increase in the maximum crucible size taking place from the 14th century, and hypothesised that this might be related to the improvement in the material properties of the pastes stemming from the introduction of grog tempering. After the analysis of a wide range of post-medieval technical ceramics, it now seems that the use of grog was rather limited. Grog tempering may have been more labour consuming, particularly as crucible making became a specialised job. If the crucibles were made by the users themselves, then these would probably have broken crucibles at hand that could easily be turned into grog – although taking care of not including slag or other fluxing elements in the fabric of new vessels. If, instead, crucibles were manufactured by specialised potters at a larger scale, the attainment of grog would require either processing wasters, liaising with the users to obtain their discarded vessels, or its artificial production by firing and crushing bricks, as described in some of the sources. In the latter case, the use of sand temper would have seemed more cost-effective. A way of, to some extent, overcoming the lack of high-fired grog in the crucible fabric may have been a high-temperature prefiring for the whole crucible (see below). Overall, it seems that the divergence in the material properties between grog- or sand-tempered crucibles would not have been dramatic, and the choice of either option may have been led by *ad hoc* factors. The example of the use of grog as a way of saving sand was already mentioned (section 6.3.2). A different yet illustrative instance would be that of the crucibles from Rio de Janeiro, where grog was used as a strategy for recycling the scarce and expensive graphitic fabrics (section 7.3.3). In any case, it should not be forgotten that there are 'one hundred ways to skin a cat' (Sillar and Tite 2000), and often crucibles may have been made simply following a known procedure without questioning it (cf. Lemonnier 1986: 165), or perhaps following specific requests by the customers.

An interesting recipe for home-made crucibles is given by the 17th-century chymist Gabriel Plattes. In an example of the resourcefulness often required in a laboratory, he suggests the use of crushed 'Flanders jugs', perhaps stoneware, as grog, together with other raw materials:

> "Take right *Flanders* Iugges, such as they usually put Bottle-Beere in, beate them to fine powder, and searce them fine as Meale: Take of this meale four pound, of the fine powder of *Tobacco* pipe Clay one pound, temper them together with the red fattish water that issueth out of an Horse Dung-hill, beate it strongly

upon a broad board with a Rowling-pin, till it be stiffe Paste, then fashion your Pots upon a peece of wood, turned like a Toppe." (Plattes 1639: 19).

The use of crushed graphite as temper is not recorded until the late 18th century (see section 7.2). Other tempering materials are mentioned in some sources, although they have not been identified archaeologically. Of particular interest appears the *ferri squama* indicated by Agricola and mentioned above (section 9.3.3). Along similar lines, Biringuccio states that the clay used for crucible making should be mixed with "an eighth part of finely ground and sifted iron scale, and also some young ram's-horn ashes" (Smith and Gnudi 1990: 391). If these suggestions truthfully reflect common practice, they can only be technically justified as an attempt to foster the vitrification of the paste during firing, in a process similar to that occurring with the feldspars melting within the fabrics of the crucibles in Oberstockstall (section 6.2.2), or that deliberately pursued by John Dwight when adding crushed glass to the clay mix (Freestone 1991). However, the addition of iron to the paste would seem at odds with the purposeful avoidance of red, *i.e.* iron-rich, clays, noted above.

Once tempered, the paste would be well kneaded and thoroughly homogenised, a process known to require several days, at least in the case of Hessian wares (Stephan 1995: 22). The next step would be the forming of the crucible.

10.2.3. Crucible forming, drying and firing

When it comes to the forming techniques, a significant contradiction is patent between historical and archaeological information. Out of all the 16th-century written sources consulted, only Biringuccio refers to the fabrication of the vessels on a potter's wheel, "just as plates are made" (Smith and Gnudi 1990: 391). Conversely, the *Probierbüchlein*, Agricola and Ercker describe the forming of crucibles and scorifiers using wood or brass moulds (Sisco and Smith 1949: 111; Hoover and Hoover 1950: 230-231; Sisco and Smith 1951: 25, 180 and 204) (figs. 188-189). The picture obtained from archaeological sources is different, in that most of the crucibles and scorifiers examined show clear signs of wheel throwing such as rotation marks on the base or horizontal striations on the body. In this sense, we could conjecture that most Renaissance treatise writers described crucible making in moulds because this is what they saw in metallurgical workshops, in the absence of specialised potters. In later written sources (Plattes 1639: 19; Gatterer 1790, as cited in Stephan 1995: 23), the use of the wheel

seems the common practice, which may be related to the specialisation and the increase in the scale of the production of technical ceramics, since wheel throwing would allow a much faster manufacture. Nonetheless, a 17th-century advocate of domestic crucible making, Johann Glauber (1651: 315ff), states that crucibles "cannot be formed by the usual art of the potters", but should be carefully shaped on very smooth moulds. This instance provides yet another compelling example of the pertinence of combining all the available sources of information if more accurate reconstructions of past technologies are to be achieved.

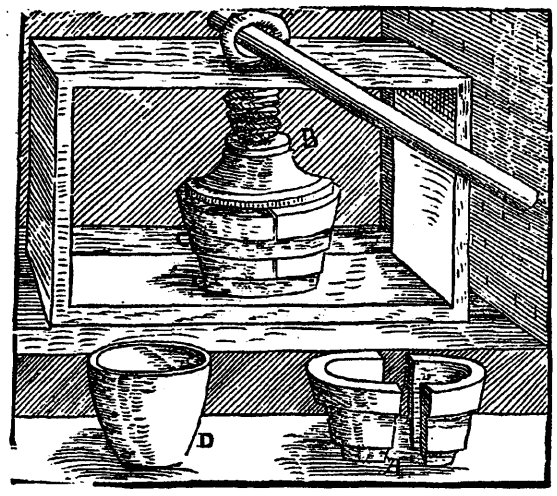


Figure 188. Mould and press for making crucibles, according to Lazarus Ercker (Sisco and Smith 1951: 181, fig. 26). A: The lower part of the wooden mould; B: The entire mould, showing the crucible shaped under the press; C: Iron hoop; D: Finished crucible.

After primary forming and subsequently finalising the shape – for example folding the rim into a triangular plan –, the vessels would be left to dry. In the 18th-century description of crucible manufacture recorded in Hesse and mentioned above (section 8.2), the importance of this stage appeared emphasised to such an extent that, in trying to avoid unwanted moisture, crucibles were primarily made during the summer season. Another author appears less exigent, and simply advises to "set them [the crucibles] to drye in your Chimney Corner a day or two" (Plattes 1639: 19). Glauber (1651: 316) recommends drying the crucibles "first in the aire, then by the heat of the fire, or fum". It would be after some drying that the crucibles could be turned up-side-down for stamping. In the case of the dark vessels, this would probably be the moment of wiping them in order to ensure a smooth and lustrous finish[37].

[37] According to 19th-sources, however, this effect was achieved by applying a graphite mash and rubbing the crucible surface after firing (Bauer 1983: 33) – which stresses the importance of appearance, perhaps as an inheritance of earlier times (see section 6.2.3).

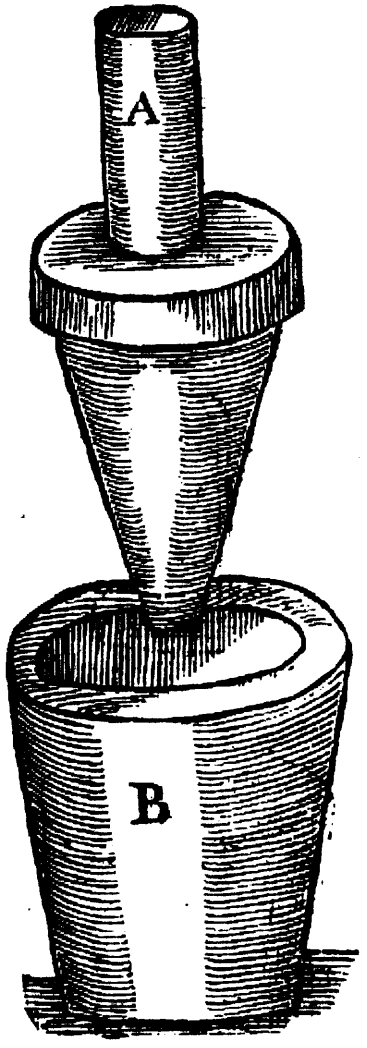


Figure 189. Mould and pestle for making crucibles, as depicted by Johann Glauber (1651: 317).

Different procedures could be followed in order to fire the crucibles. This stage of the *chaîne* may have constituted a decisive – yet underresearched – factor affecting the formal and material properties of the vessels and, by extension, their perception, consumption and performance. According to Biringuccio, crucibles could be "baked in furnaces like pots or other vessels" (Smith and Gnudi 1990: 391). This could well be the case of the 'alternative' crucibles found in London and Cologne discussed above, which show no or very limited vitrification. In this, they are analogous to earlier crucibles, for which a link between crucible manufacture and the established pottery industry may be suggested (Freestone and Tite 1986: 50; Freestone 1989: 159). Imaginably, when crucibles were made by the users themselves, they would be fired directly in the metallurgical, assaying or 'philosophical' furnace. Gabriel Plattes describes a process whereby the previously unfired vessels are placed in the furnace only when you need to use them, but you "set them in the fire at the first kindling: and so let the Fire steale upon them till they be red hot: [only] then put in your Mettle and ingredients" (Plattes 1639: 19-20; see also Glauber 1651: 316).

Finally, and in stark contrast, the manufacture of both the famous Bavarian and Hessian crucibles involved the use of specialised firing techniques. For the dark crucibles, this involved a moderately reducing firing and culminated by choking the kiln with organic material in order to produce the desired black lustre. In Hesse, large, purpose-made kilns were used to fire the vessels in extremely hot and oxidising conditions, perhaps borrowing a technique used for stoneware[38]. Moreover, it does not

[38] The early connection between stoneware and crucible firing techniques remains to be further investigated. In the 17th century, this connection is suggested by Glauber (1651: 313-317) and even illustrated by Dwight, who manufactured both stoneware and technical ceramics (Green 1999).

seem coincidental that these are recurrently the crucibles showing higher firing temperatures. In spite of their superior refractoriness, the vitrification observed in the samples of unused vessels from both Oberstockstall and Hesse is invariably greater than that of other crucibles – advanced initial to extensive in those from Oberstockstall, and complete vitrification in the Hessian ones (see below).

In the author's view, those vessels which were carefully high-fired in the crucible maker's kiln may have been less likely to crack when re-heated in the furnace. Furthermore, a more vitrified ceramic matrix would be more impervious, hence holding liquids more efficiently but also offering more resistance to corrosion. An additional advantage would have been a higher tensile strength. Allegedly, the technical drawback might be a higher brittleness, since the glassy matrix would be more prone to develop fissures upon thermal or mechanical shock (Kilikoglou *et al.* 1998; Tite *et al.* 2001). Nonetheless, this flaw could have been countered by the presence of temper and other nonplastic inclusions that would arrest and scatter lines of fracture before they became catastrophic. Unfortunately, at present we lack relevant experimental and analytical data to confirm this hypothesis. The most systematic studies on the material properties and behaviour of different ceramics under assorted stresses have concentrated on ordinary vessels such as cooking pots or containers (Kilikoglou *et al.* 1998; Vekinis and Kilikoglou 1998; Tite *et al.* 2001; Tite and Kilikoglou 2002; Kilikoglou and Vekinis 2002). Technical ceramics, however, pose specific questions, for example those deriving from the fact that they would frequently be used at temperatures above those reached in the original manufacture, together with the specific chemical and mechanical strains to which they would be subjected.

Even if lacking supportive experimental measurements, this proposition, that is the decisive importance of a long, high prefiring, finds support on the experience-based observations of a lucid mind with notorious expertise in making and using crucibles: Johann Glauber. Confronting the sightless belief of his contemporaries, and even modern suppositions, Glauber played down the role of renowned clays and emphasised that the key to a proficient crucible was a good, strong, prefiring. He was bold enough to assert that anyone could make crucibles as good as the famous Hessian and Ipsian ones without any need of Hessian or Ipsian clay, but provided that the manufacture and the firing were appropriate. Some of his statements deserve full mention:

> "Chymists have been in a great error a long time, and not only they but also goldsmiths, and they that separate metals, as also others that need the help of crucibles, who perswade themselves that the best earth that is fit to make the best crucibles is to be found no where but in *Hassia*; and therefore with great charges have caused that Gibsensian crucibles be brought over; not considering that almost in every place in Germany such earth is to be found [...].
> But although this earth be brought from thence to other places, yet such strong crucibles could not be made thereof, the cause whereof being not the constitution of the aire, and place to which some have falsly imputed it, but an error in the making and burning of them [...].
> But now experience has taught us that any good earth doth become stony in a violent fire, without respect of the place where it is taken. Wherefore it is very probable, being a thing possible, that such vessels are made els where: for every earth being burnt retaining a white colour, *viz.* with an indifferent fire, makes pots, and crucibles porous, but with a stronger, and with a longer delay, compact like glasse [...]." (Glauber 1651: 313-314)

In any case, we should not forget that a high manufacturing firing would also result in a crucible offering a better 'ring', that is, the emission of a longer, deeper sound when struck with a finger nail. This directly links with the perception of the crucibles in the market and, ideally, the achievement of the main goal of the crucible makers: their purchase by the customer.

10.3. Perception and choices in the consumption of crucibles

Two of the assemblages addressed in this thesis, namely those from the Cripplegate Buildings and the Old Ashmolean Laboratory, contain crucibles that were produced in more than one location. In the case of Cripplegate, some of the technical ceramics have been identified as Hessian (section 8.3.1), whilst others seem to have been produced locally in London (section 9.3). In the Old Ashmolean, most of the crucibles were ascribed to Hesse (section 8.3.1), but at least one came from another producer: the graphitic crucible from Obernzell (section 7.3.2). In both instances, the remains were found in waste deposits whose contents, even if dumped at once, may have been accumulated over relatively long periods, therefore we cannot safely affirm

that the crucibles from diverse origins were being used simultaneously. This suffices, nonetheless, to indicate that technical ceramics from different manufacturers reached, in some cases at least, identical destinations. In other words, bright, dark, and other crucibles confronted each other in the market and, possibly, in the laboratory[39]. The archaeologist's curiosity is inevitably sparked off: on which basis would one crucible or another be selected in each case? Which criteria would be leading the choice?

It is not necessary to stress that in the past, like in the present, different people acquired and used the same items with diverse motivations, expectations and purposes. Since human decisions are ultimately directed by humans and their circumstance, our only path to understand past choices will be the investigation of the context where they were made. It follows that only detailed analysis of case studies may serve as a basis to interpret human intentions when choosing and using a particular tool. A preliminary attempt was performed with the Oberstockstall assemblage, trying to relate the different tools and their fabrics to each other, to differing traces of use and to the wider archaeological, political, social and cultural context. The same cannot be attempted for the other case studies within the framework of a single PhD project. For the time being, however, it is possible to explore the various criteria that may (or may not!) have been at stake when a given chymist chose one crucible type or another. Even if not definitive answers, this global perspective may provide a guide for future work.

To start with, one of the foremost criteria that may have applied is cost. Imported crucibles were probably more expensive than locally made alternatives, and this explains a meeting of the Royal Society of Arts in London, in 1755, seeking to promote the manufacture of crucibles from British materials (Cotter 1992: 268). Before acquiring or using an expensive vessel, it would have been reasonable to estimate the profit (or loss) to be obtained – surely, a gunmetal cauldron would not foreshadow as much benefit as the philosopher's stone, and its production would hence attract lesser investment. Put in other words:

> "For not all & every crucible can alwayes and every where be made equal, and be of a like durableness in the fire, though they are made most diligently: and

[39] Against this supposition, it could be argued that alternative crucible types were available at different times, depending on specific junctures of trade and supply. However, by the 16th century if not before, there are established international transport and commerce routes channelling, for example, stoneware. Thus it seems plausible that if a chymist working in London or Oxford wanted to purchase, say, a Hessian crucible, this would be feasible.

therefore a consideration being had of their goodness they may be used for divers uses, *and the better may be used in the melting of the better metals*" (Glauber 1651: 320, my emphasis).

Related to this, the technical requirements and expected performance could be considered. Here, issues such as previous experience, the reputation of different productions, or simply habit or transmitted knowledge, would play a role (see below). For example, from a modern utilitarian viewpoint, we could conjecture that a brass maker would require a lidded or relatively closed crucible, in order to prevent 'colour' – *i.e.* zinc – losses in the fumes, whilst a chrysopoetic chymist would need an especially refractory vessel, given their emphasis on purifying substances by heating and calcining them to the utmost. An artisan conducting reactions involving salts and fluxes, such as an ore assayer or a 'philosophical' gold maker, would normally be more concerned with the resistance of the vessels to chemical attack than an ordinary copper-alloy melter and caster. If the crucibles were to be used many times, then sturdier, more wear-resistant and non-absorbent fabrics would be needed. A late 18th-century author, for instance, notes that black, Ipsian or Passauer, crucibles, withstand thermal shock better and *more often* than Hessian wares (Beckmann 1780, as cited in Stephan 1995: 31; Beckmann 1787, as cited in Bauer 1976: 14-15). With repeated uses, microcracks generated by steep temperature changes would expand until the crucible failed. Nevertheless, this would not be such a crucial factor if the vessels were to be discarded after the first use – for example, to prevent cross-contamination in metallurgical analysis. On the contrary, in Beckmann's opinion, Hessian crucibles are more resistant to corrosion by "metallic glasses".

For certain purposes, crucible users may have preferred to persevere in the use of one vessel type in order to keep variables constant in their experiments, and/or to stick to the utensil whose behaviour was known and controlled. Thus the underlying factor would be reproducibility or, to use Glauber's (1651: 320) term, "certainty sake", a concept inherent to the development of modern science (Newman 2000; cf. Principe 2000). This protocol is explicit in Norton's verses:

"Consider all Circumstances, and set your delight
To Keepe Uniformity of all things Requisite.
Use one manner of Vessell in Matter and in Shape,
Beware of Commixtion that nothing miscape." (Norton 1652: 49)

If, as it seems likely, people judged the potential behaviour of a crucible before acquiring and using it, then it is worth asking: how would they envisage that performance prior to use? Paraphrasing Glauber (1651: 320), how would the "consideration of their goodness" be executed? What would tell a chymist that *this, and not that*, is the appropriate crucible? The question necessarily brings us back to discuss the perception of the crucibles. A crucible would be assessed by looking at it, perhaps handling it, touching and inspecting its surface, listening to its ring, maybe turning it to glance at a stamp (cf. Sillar 1997). This apprehension would be subjective and influenced by previous knowledge and experience, habit, reputation, the need of replication... expectations that could only be confronted with the external formal properties of the vessel[40]. On this foundation, as discussed earlier (section 6.2.3), the smooth, black lustre of the Bavarian crucibles acquired a special significance: it added cohesion and identity to all the vessels belonging to the same group. This argument is reinforced when examined from a wider perspective.

In Renaissance and later Europe, two major producers compete for the market of technical ceramics. The crucibles from both sources are fine: in general terms, and from a present-day standpoint, either type would serve any common utilitarian purpose with more or less equivalent efficiency. They are, however, radically different: one producer sells crucibles that are invariably dark and smooth; the other one offers bright, pimply vessels. In this picture, the peculiar appearance not only strengthens the ties amongst the constituents of the same group, but also accentuates their disparity with regards to the others. When confronted in the market, appearance and texture would be the most conspicuous factors telling a chymist that *this, and not that*, is the desired vessel. Remarkably, the 18th-century comparison between Hessian and Bavarian crucibles mentioned above starts by noting that the former are yellow-grey or reddish, whilst the latter are black (Beckmann 1780, as cited in Stephan 1995: 31; Beckmann 1787, as cited in Bauer 1976: 14-15). The very existence of a contrasting opposite would make it easier for the consumer to identify the "reputable pot", whichever this may have been (Sillar 1997; Sillar 2000b: 79-80). This 'reputable appearance' might convey different meanings in each case: it could mean provenance, quality, tradition, adherence to an authoritative recipe or any other combination of incentives. On most occasions, however, this causal

[40] Alternatively, crucibles may have been ordered from known suppliers, but also in this case the most characteristic features to be considered would be the outward appearance and/or the provenance of the vessels, rather than their material properties.

relationship between signifier and meaning would not even operate; nonverbal, learned, apprehended, ritualised factors would lead the choice (Pfaffenberger 1992). People would choose a bright, pimply crucible because they 'needed' a bright, pimply crucible, because that is the way things are done or, perhaps, because that is the way things have *always* been done (Lemonnier 1986: 165). This idea is epitomised by the case of the dark crucibles, which clearly *had* to be black. We could even argue that 'black' did not mean anything but 'black'.

Colour is increasingly addressed in archaeological studies as a key constituent of the materiality of artefacts, playing a crucial role in the perception and categorisation of material culture (Jones and Bradley 1999; Jones and MacGregor 2002a). What people saw with their naked eyes is often more meaningful than what we can see under a microscope. These innovative approaches to archaeological artefacts usually consider colour as either a symbol or a signifier, probably because this is how we tend to conceive colours today: for example, red ochre symbolises blood (Scarre 2002: 228-230; Jones and MacGregor 2002b: 7-9), or the green shade of a stone axe signifies that it comes from a particular source (Cooney 2002). Nonetheless, the discussion above illustrates that colour may be taken as a symptom: rather than arguing that a given axe was preferred because of its source, as indicated by its colour, one could claim that a green axe was chosen because of its very colour – 'it is good because it is green'. Taking the argument further, it might be suggested that ochre and blood could be categorised as the same entity: redness would make them equivalent. Similar principles may have applied in the perception and selection of crucibles.

From an analytical standpoint, it has been useful to separate out the formal properties, the material properties and the performance characteristics of the technical ceramics (for discussion of these terms, see Schiffer and Skibo 1997; Skibo and Schiffer 2001; Sillar 2003; Schiffer 2003, and section 3.6). On this basis, it would be tempting to construct a model of consumption whereby the crucible user would assess the formal properties of a vessel, infer from them its material properties and, on this basis, visualise its performance characteristics. Such an analytical model involves some bold assumptions, though. Firstly, we cannot assume that past peoples separated out these categories as we do today. As noted by Shanks and Tilley (1992: 106), "'The things themselves' (i.e. the data on which we work) exist only in so far as they as conceptualized". Secondly, thinking in these terms compels surmising a cause-effect logic leading to the decision, from what one sees – e.g. triangular and smooth – through

what one understands – e.g. good for pouring and corrosion resistant – to what one expects – e.g. an efficient service in assaying silver ores. It takes for granted a linear mode of ruminating: 'the pot gives a good ring because its fabric is hard, and therefore it will perform well'. In actual fact, as discussed above, consumption may have worked as a much more complicated network of factors, moved by perceptive stimuli that cannot be explained in a logical sentence because neither are they logical nor can they be verbalised. "The portion of technical knowledge that people can verbalize represents only the tip of the iceberg" (Pfaffenberger 1992: 508) and, owing to this, Renaissance treatises alone will never explain Renaissance technological choices in full.

The limitations of a rigid model of consumption may also come to light if we return to the stamp exhibited by some of these crucibles, and try to assess its influence in the users' choice. At a practical level, the presence of a particular stamp would help categorise a crucible as an element of a group. It could be necessary, for example, to tell apart a dark Ipsian crucible from a dark Obernzeller one. Nonetheless, taking into account the wealth of concepts purported by the '4-shaped' stamp of the latter, and the complex technological, social and cultural network where it is inscribed (see section 7.4), we might have to be prepared to accept that a crucible would be expected to behave in a particular way in the furnace *because of* that stamp, or even, that the crucible would be bought *because of* the stamp, as a matter of cultural allegiance, irrespective of its effects (if any) on the vessel's high-temperature performance. In any case, these crucibles would perform, amongst others, a very important pursuit: the transmission of ideas.

10.4. Crucibles in their sociotechnical system: the keys to success

From the discussion above, it should be clear that we cannot decide that one crucible is better or worse than another in absolute terms. That would be like asserting that a cannon is a better weapon than a rolled newspaper – before having to confront a mosquito. The reality is, however, that some crucible types, namely the bright and the dark ones, progressively took over the market at the expense of alternative productions. Why did that happen?

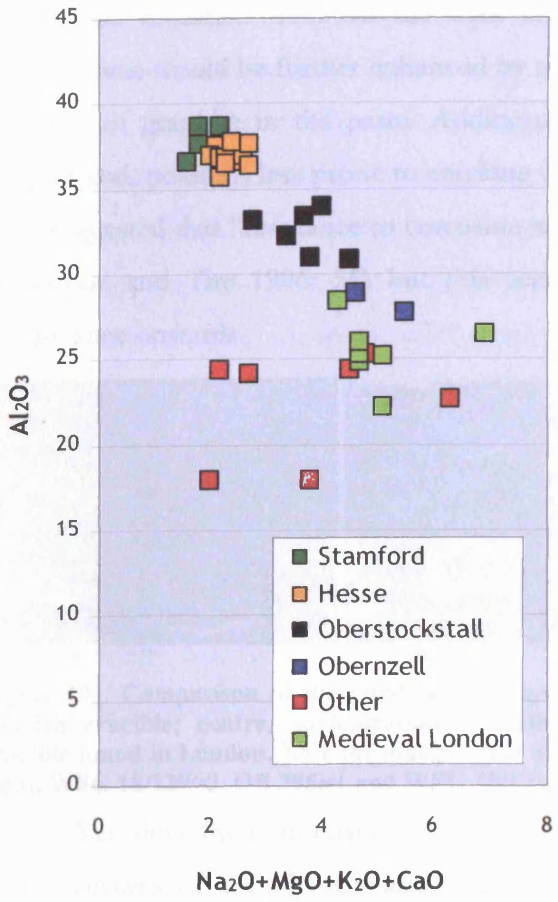


Figure 190. Comparative graph of the thermal refractoriness of the ceramic matrices of different medieval and post-medieval crucibles discussed in this thesis. While heat resistance increases with higher alumina levels in the ceramic matrices (vertical axis), it decreases with higher alkali and earth alkali concentrations (horizontal axis). Note that the most famous post-medieval crucibles tend to show higher alumina levels, but the medieval Stamford wares appear the most refractory ones (data for Stamford and Medieval London crucibles from Freestone and Tite 1986: 42).

A sensible starting point to resolve this question may be to compare all the Renaissance crucibles analysed within this thesis, and try to identify singularities in the successful ones. One factor will immediately crop up: thermal refractoriness. Invariably, the chemical compositions of the ceramic matrices of both bright and dark crucibles show the high alumina, and relatively low alkali and earth alkali, concentrations, which are characteristic of highly refractory ceramics. This feature alone does not appear a sufficient explanation, though, if we widen our scope. In medieval England, the potters from Stamford produced considerable amounts of crucibles that were practically as refractory as the Hessian ones (fig. 190), however this industry never reached the expansion of its famous German counterpart, and declined in the 14th century (Kilmurry 1980). Furthermore, refiring experiments of other crucibles have shown that, even if not as refractory, they would be refractory enough to remain stable up to 1200 °C (Freestone and Tite 1986; Bayley *et al.* 1991).

While not disdaining thermal refractoriness, another singular feature in the successful crucibles may be identified: the resistance to chemical attack. Here, bright and dark crucibles – especially the former – do stand out, with no comparable competitor (fig. 191). As argued above, the high-temperature prefiring conferred these vessels a more vitrified – hence more corrosion resistant – ceramic matrix. In the bright crucibles, the dense and vitrified matrix would compensate for the presence of cracks

due to the abundant sand. In the dark ones, even if not as glassy, the chemical refractoriness would be further enhanced by the especially smooth finish and, often, the presence of graphite in the paste. Additionally, high fired crucibles would be more resilient and, possibly, less prone to cracking in the first use. For earlier crucibles, it has been suggested that "resistance to corrosion was not a major concern of the craftsmen" (Freestone and Tite 1986: 55) but this seems to be no longer the case from the Renaissance onwards.

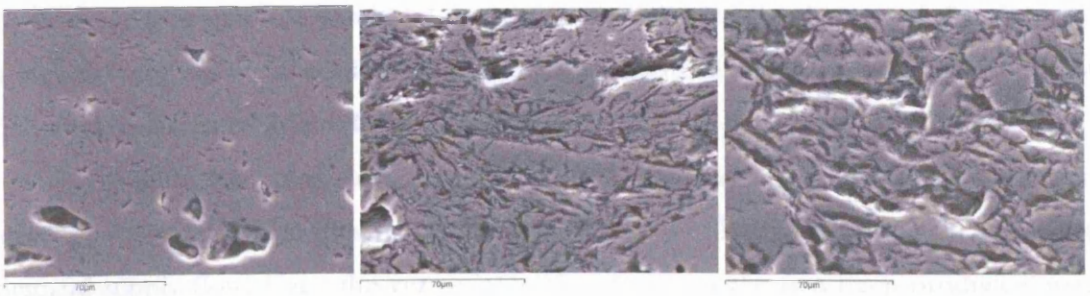


Figure 191. Comparison of the vitrification stages of different crucible matrices. Left, bright Hessian crucible; centre, dark graphitic crucible from Oberstockstall; right, 'alternative' crucible found in London. Note the much higher vitrification of the Hessian matrix (from left to right, WFG 18/129/s2, OB 395/s1 and WFG 18/121/s1; all SE at 800x).

Yet, does the combination of thermally *and* chemical refractoriness explain, *per se*, the success of the Hessian and Bavarian crucibles? No, unless we consider the changing demand. It is not a coincidence that the growth of these industries runs parallel to – or, rather, intertwined with – the expansion and modification of pyrotechnological practices across Europe. As discussed in the first chapters, the Renaissance witnesses an acme in the quest for the philosopher's stone, together with developments in the mining industry that require the recurrent conduction of ore analyses, and in addition to a widespread interest in investigating nature. These are strands of a wider cultural and economic atmosphere, all of them recurrently necessitating high-temperature, small-scale, carefully controlled, reproducible experiments such as fire assays, and other reactions using newly discovered acids and fluxes. Spagyric chymistry, concerned with the decomposition of substances into their Essentials by means of chemical attack and extreme temperatures, may have played a special role here. Just as these reactions would attack the sample processed, so would they corrode the vessel serving as a container. Only thermally *and* chemically refractory crucibles could serve these new needs and, insofar as we can say that these crucible making industries developed thanks to the existing demand, we can also assert that the

Renaissance developments in metallurgical analysis were only possible because these technical ceramics existed (Martinón-Torres and Rehren 2004b).

This change in crucible utilisation and, relatedly, in crucible design, is observed by Glauber (1651: 313), who clearly notes that most crucibles are valid for holding metals, but few can cope with molten "salts". Having much lower surface tension than liquid metal, these salts would be more prone to wet a permeable fabric. Smooth, vitrified surfaces, large inclusions with relatively small surface areas or chemically inert particles such as graphite would be the means to tackle this problem and the chemical attack by salts and metal oxides. The required precision, accuracy and reproducibility of chemical reactions could not be ensured otherwise.

Natural resources, economic activity, technical know-how and cultural atmosphere converged in these particular areas of Central Europe creating the necessary demand-supply flow. Even the very competition between the two main producers may have served as a further spur. Still, these factors alone might not have lead to such an industrial expansion had it not been for the appropriate political support. Both in Obernzell and in Großalmerode, during the 16th and 17th centuries and probably earlier, different laws governed and protected the exploitation of clay deposits, and the manufacture and commerce of crucibles, favouring certain dynasties or guilds. Significantly, characters of political relevance such as Count Moritz in Hesse (chymist and holder of several such privileges) or Urban von Trenbach in Passau (archbishop and chymist working in Oberstockstall) would be themselves interested in an adequate supply of technical ceramics.

It is impossible to know whether these industries would have flourished without such institutional assistance, but it seems plausible that the advantageous conditions provided a favourable background for the market expansion. With the certainty of the market domination, these crucible makers would have been able to specialise and standardise their productions, as well as to develop firm, efficient and cost-effective channels for the distribution of their goods.

In sum, the success of the dark and bright crucibles can be seen as the success of an entire "sociotechnical system" (Pfaffenberger 1992). It required the synchronisation of a complex network involving natural elements, technical skills and coordination of labour, social customs and cultural conceptions, linguistic and nonverbal forms of perception and communication, and a suitable political and legal framework. The establishment of these crucible making industries is the expression of an intricate

activity system with such apparently disparate constituents as clay, kilns, mines, archbishops and counts, gold, guilds, privileges, the Renaissance understanding of nature and the quest for the philosopher's stone. Once material, social and ritual dimensions were harmonised in a comprehensive manner, this technological system could not but succeed[41].

By the time privileges were repealed and growing competitors emerged across Europe, bright and dark crucibles were already established. The techniques were widely available, since crucibles did not require any more sophisticated know-how than stoneware making, but the sociotechnical system was working and it would not be easy to break it (Pfaffenberger 1992: 498-502). New factors were sustaining it. In the mid 18th century, Diderot (as cited in Cotter 1992: 265) observes that the quality of French crucibles is comparable to that of the more successful productions, but he must concede that "German crucibles, and particularly those of Hesse have long been famous amongst chemists of all nations". For centuries, these crucibles had been mentioned in metallurgical treatises and in the most authoritative recipes for making the philosopher's stone. They had been widely used and tested favourably. They had an identity, a reputation, a history, perhaps a meaningful stamp. Production and consumption were now ritualised (cf. Pfaffenberger 1992: 501-510). Arguably, there was no point in trying alternatives or, if there was, the possibility may not have been given a thought.

10.5. Summary

The historical, archaeological and analytical study allows a preliminary approach to the production and consumption of crucibles in post-medieval Europe, with a special focus on the choices made by manufacturers and end users, and the special role played by perception in decision-making.

Clays for crucible manufacture were selected on the basis of sensorial criteria, primary colour, and it seems that red clays – *i.e.* those richer in iron oxide – were avoided. From a technical viewpoint, the only explicit concern regarding the clay and its

[41] Even though I find Pfaffenberger's 'sociotechnical system' a very useful concept, I should note my concerns with his Marxist interpretation of the system as a network of facts and values primarily orchestrated by those in power in order to legitimate and perpetuate the social order. In this sense, his view is akin to the argument presented by Shanks and Tilley (1992: 133). I do not think that the success of some crucible productions was only due to a deliberate effort on the part of those manufacturing them or somewhat benefitting from this industry. Rather, I accept a significant level of contingency, free will and even chance in this historical development.

resulting properties was its thermal refractoriness, even though the analysis of the crucibles indicates that these would have had to meet many other strains. The clay was usually tempered with sand and, occasionally, with grog. Archaeological examples of Renaissance and later technical ceramics suggest a preference for quartz sand, but either alternative does not seem to have a decisive effect in the performance of the vessels, and their choice was probably led by specific dynamics of each technological context. After refining and homogenising the clay mixture, the crucibles would be formed in moulds or on a potter's wheel. Seemingly, moulds were primarily used for *ad hoc* productions by the users themselves, which explains their recurrent appearance in the written metallurgical and chymical sources, contradicted by archaeological information.

Frequently overlooked by early modern writers as much as by present-day researchers, the firing of the crucibles may have had a crucial importance in the production of the successful bright and dark vessels. Specific firing techniques and temperatures would result, on the one hand, in their peculiar appearance, and, on the other hand, in highly vitrified ceramic matrices that would offer more resistance to wetting and chemical attack.

The attention to crucible making paid by some treatise writers suggests that the crucibles may not have been readily available always and everywhere. Archaeology and written sources, however, prove the long-distance trade of both dark and bright crucibles to the same locations, and their use in equivalent activity contexts. Clearly, the reasons behind the choice of one crucible or another were not universal, and they require detailed investigation in each case. In acquiring or using a crucible, the user would probably bear in mind the intended utilisation but, on a routine basis, this would not necessarily involve an assessment of the performance characteristics of the vessel. Economic and utilitarian motivations, but also habit, reputation, cultural bonds, transmitted knowledge, scientific concerns and other contextual factors would be at stake. The specific appearance of the different crucibles would appear as a materialisation of the user's needs, but these would vary and may not even have been verbalised.

In explaining the success of the bright crucibles from Hesse and the dark crucibles from Bavaria and surroundings, the whole sociotechnical system ought to be considered. Both crucible types, although different in their design and appearance, showed an exceptional resistance to chemical and thermal stresses. Their technical quality responded to, and enabled, the changing needs of different pyrotechnological

activities such as ore assay, chrysopoeia and chemical investigation. These required large numbers of standard vessels capable to withstand aggressive chemicals at extremely high temperatures, and they did so because of renewed worldviews and expectations, social engagements and economic enterprises. At the same time, the production of such crucibles could only be ensured by a combination of natural resources, political protection, market competition, technical advance, infrastructural development and increasing demand. The successful crucibles were just as necessary as all the other entities harmonically engaged in the same network. The functioning of the sociotechnical system facilitated the ritualisation of production and consumption and, together with it, its perpetuation.

11. Conclusions and further work

"The past and the present are within the field of my inquiry, but what a man may do in the future is a hard question to answer."
Sir Arthur Conan Doyle (1859-1930)

"The true worth of an experimenter consists in his pursuing not only what he seeks in his experiment, but also what he did not seek."
Claude Bernard (1813 - 1878)

"Serious work on post-medieval crucibles has scarcely begun."
John Cotter (1992)

The Renaissance was a period of fundamental changes in the ways people conceived themselves and the surrounding world. The departure from tradition was long and multifaceted, and indeed it was coupled with continuities in knowledge and skill that simply moved from different spheres of activity into the academic discourse. Thus any general statement about this time is at risk of oversimplifying a complex reality. The work presented in this thesis largely concentrated on a very small sample of the Renaissance world: the laboratory in Oberstockstall; more precisely, the technical ceramics used there for high-temperature reactions. This study, however, not only served to investigate specific issues pertaining to the intra-history of Oberstockstall, but also wider components of the context in which the laboratory equipment, and their makers and users, were inextricably embedded.

As the study expanded to incorporate technical ceramics from other regions, periods and contexts of utilisation, it has been possible to construct a preliminary picture of the production and distribution of crucibles across the post-medieval world, their properties and utilisation. This has proved informative with regards to the transfer of tools, ideas and skills amongst different spheres of activity, as well as the perception of matter and the choices made within an evolving sociotechnical system.

This chapter summarises the main conclusions of the work carried out so far, together with some lines for future developments.

11.1. Technical ceramics in the Oberstockstall laboratory

The ceramic equipment recovered in Oberstockstall shows a remarkable degree of standardisation and specialisation. The different vessel types are coherent in their design, generally showing the formal and material properties necessary for their specific practical utilisation. Whether they were produced by potters specialised in technical ceramics remains to be investigated.

Several groups of ceramic fabrics could be demarcated on the basis of macroscopic, chemical and mineralogical analyses. The main group is formed by a relatively narrow range of fabrics used for crucibles and scorifiers, but also for other vessels such as black or orange-glazed ceramic containers. Some variability within this group is noticed, particularly in the use of grog as temper for some of the scorifiers or in the restriction of graphitic clays for equipment to be used at high temperatures. A second group is constituted by the stamped black wares obtained from the village of Obernzell, in Bavaria. It is interesting to note that the high-temperature ceramics used in Oberstockstall were not from that producer, despite the reputation of the Bavarian crucibles and the fact that one of the chymists plausibly working in Oberstockstall spent some years living in Obernzell as archbishop of Passau. Specialised ceramics used for operations such as sublimation and distillation fall outside the previous fabric groups and suggest a distinct provenance, hinting further at the existence of specialised potters trading their goods across wide distances.

Focussing on crucibles and scorifiers, it was shown how their formal and material properties were generally satisfactory for the technical stresses to which they were subjected, but also that their users knew how to adapt the conditions of utilisation in order to minimise risks of failure. This is illustrated, for example, by the use of crucible bases made from less refractory clay, but also by more subtle details such as the fact that graphitic vessels were used at the highest temperatures only in reducing atmospheres, where graphite inclusions would not burn out. In the same vein, scorifiers were employed as an easily accessible resource that allowed savings in the less available ash-based cupels, but this required dexterity and swiftness on the part of the chymist – indeed, it is in the use of scorifiers where more failures are documented.

While these practices exhibit an acquaintance with technical knowledge acquired by experience that resembles present-day conceptions, other factors bring us back to the Aristotelian understanding of matter that would only be abandoned in the wake of the

Renaissance scientific discoveries. A paradigmatic instance of this is offered by the fact that as much importance was given to the appearance of the technical ceramics as to the composition of the raw materials employed or the manufacturing technique: seemingly, the black and lustrous look of the triangular crucibles was more imperative than the graphite content of their fabrics, which would explain the presence of some black, non-graphitic crucibles in the assemblage. The crucial role imparted by Renaissance peoples to the immediately sensorial dimensions of nature has become apparent in other parts of this thesis such as the historical recommendations given for the selection of clays or the production of the philosopher's stone.

The cupels tender a counterpoint to the quasi-industrial standardisation of the other pieces of equipment in the assemblage. Their atypical manufacture in the laboratory, involving the debasing of bone ash with clay and the partial recycling of used cupels, illustrates the versatility of laboratory practices whilst denoting a limited understanding of the reasons to choose bone ash for cupellation. Furthermore, the apparent lack of specialised producers of cupels is rather astounding, particularly if we bear in mind their widespread use since late medieval times. This may be related to their fragility, which complicated their transport.

Future analyses of the slag and other residues within the assemblage will furnish more details of the reactions carried out in this laboratory, and the corresponding comprehension of natural elements. On the basis of the work so far, the laboratory can be ascribed to the broad realm of fire assay and/or refining of noble metals. The operations would often involve a reduction stage of an ore sample in a triangular crucible collecting any noble metals in a lead regulus, followed by the concentration of the noble metal content by the oxidation of this lead-dominated bullion in a scorifier, and culminated in cupellation, possibly before the parting of gold from silver using mineral acids. In Oberstockstall, this sequence started in consistently black crucibles and concluded in invariably white cupels, precisely the beginning and the end of a succession of colours which, according to chymists such as Thomas Norton, were essential in the production of the philosopher's stone. However, variations of this scheme and the practice of other activities cannot be disregarded.

The final intent of the high-temperature reactions at Oberstockstall has to remain an open question. Was this experimentation for the sake of knowledge? Were these attempts at metallic transmutation? Were these ore assayers working for a mining entrepreneur? Additional analyses of the equipment and related historical sources may

provide further clues, however the fact that the same operations may have been undertaken for a variety of motives means that it may never be possible to ascribe these remains to a single purpose. At present, the location of the laboratory in a sacristy, together with the written documents attesting the economic bankruptcy of one of the priests-chymists, denote an exceptional degree of concern and motivation, whatever the pursuit. In any case, the ultimate purpose of these activities, the professional speciality of those conducting them or their related worldviews cannot be interpreted too narrowly, as demonstrated in the revised approach to the written evidence presented in this thesis.

11.2. The interpretation of Renaissance metallurgy and science

Much emphasis has been laid throughout this work on the need to avoid present-day categories and assumptions when trying to reconstruct the past. The historical review prompted by the archaeological remains revealed a substantially anachronistic bias in the traditional studies of Renaissance science. A change in our investigative approaches is encouraged, which may entail, on the one hand, concentrating more attentively on specific case studies and, on the other hand, widening the reference framework for interpretations.

For professionals dealing with archaeological remains of fire assay and related activities, it is crucial to widen the scope of historical sources of reference beyond the well-known metallurgical treatises. For both archaeologists and historians alike, we need to be aware that gold making was just one activity within the wider chymical domain, conceived as feasible by many. The complex and variable scope of Renaissance chymistry overlapped substantially with what we would nowadays call metallurgy, and trying to demarcate the 'science' and the 'craft' too restrictively will often turn out to be futile and anachronistic. Similarly, as the Renaissance entailed a diversification in the human approaches to nature, we should avoid generalising about the worldviews and expectations of different peoples based simply on the analogies among the activities they carried out. Perhaps it is time for a new turn in the history of chemistry and metallurgy, from the vast historical reconstructions of the traditional historiography to more focussed contextual studies which may contribute to a better understanding of the wider picture. This newly written history, built as a sum of different parts, requires a sound theoretical framework giving coherence to the whole. The role of well-researched

archaeological case studies within this ambitious programme is clear, and the ongoing work on the remains from Oberstockstall aspires to constitute an example.

The historical and analytical study of post-medieval crucibles from across the world reinforces these ideas about the intertwining – and divergence – of various spheres of activity, with the very same crucible types, manufactured by the same producers, used in different contexts and for disparate purposes.

11.3. Crucibles in post-medieval Europe and beyond

Since the late Middle Ages, if not before, large-scale crucible manufacturing industries developed in two small regions of Central Europe. These were respectively centred around Almerode and Epterode in Hesse (Central Germany), and Obernzell in Bavaria (Southern Germany). Their products were traded in large numbers within Europe and America, and used for a wide array of high-temperature operations, including ore assaying, coin minting, metallurgical analysis and chemical experimentation, glass melting, glaze preparation, bronze and brass metallurgy, and attempts at metal transmutation. It should be noted, however, that they co-existed for centuries with local and *ad hoc* products.

The famous Hessian crucibles were made with a very lean and refractory clay heavily tempered with quartz sand and, occasionally, grog, and prefired to very high temperatures (above 1200 °C) in an oxidising atmosphere. As a result, unused vessels generally show a bright orange colour and pimply texture. The crucibles produced in Bavaria and surrounding regions were generally manufactured with graphitic fabrics ensuring a smooth surface finish, and prefired to rather high temperatures (950-1050 °C) in a moderately reducing and smoky kiln, which conferred them a black and lustrous look.

In general terms, both crucible types showed sufficient technical quality to withstand the thermal, mechanical and chemical strains they were exposed to during use. Trying to compare their material properties in absolute terms makes little archaeological or historical sense, as it seems clear that different utilisations would result in varying stresses, and users would often adjust their use of the equipment in order to minimise failures. The information available so far shows that crucibles from both sources were used for similar purposes, sometimes within the same laboratory. Therefore, the reasons

behind the choice of either type should be sought outside their technical quality – at least, if this is understood in the narrow modern sense.

In a preliminary approach, it has been shown that the different crucibles would be chosen on the basis of variable motivations that may have involved an assessment of their performance characteristics as much as their reputation, cultural allegiances, habit and transmitted knowledge, or scientific concerns with reproducibility. The differing appearance of the 'dark' and the 'bright' crucibles, conditioning their perception by the consumer, must have been a critical factor affecting their acquisition and use. From a broad perspective, the great development of these crucible making industries was both required and enabled by a changing sociotechnical system that engaged a network of natural factors, social agents and historical junctures. This system materialised in the Renaissance laboratory, and it is not exaggerated to affirm that just as chymistry needed these crucibles, so did the crucibles need chymistry.

Hopefully, this thesis offers useful groundwork for further research on the production, consumption and distribution of crucibles in the post-medieval world. This may be greatly enhanced by a more detailed study of the two main production centres – it should not be forgotten that the reference material used in this thesis for both of them comes from uncontextualised finds – to develop a more fine-grained reconstruction of their respective chronology, variability and geographical distribution. Related to this, it will be interesting to compare the long-distance trade of Central European technical ceramics to that of domestic pottery produced in the same region, and study the role of this commerce in the configuration of a 'European culture' and its spread to colonies.

In the specific case of the dark crucibles, several issues deserve special scrutiny: on the one hand, the competition amongst different producers of very similar crucibles in neighbouring areas; on the other hand, the evolving understanding and use of naturally graphitic clays and, later, graphite-tempered ceramics. These enquiries will require a systematic and diachronic comparison of the fabrics of well-dated ordinary and technical black wares and, ideally, the development of analytical criteria to distinguish natural graphite inclusions from crushed graphite used as temper.

Another issue deserving further attention is the evocative 4-shaped stamp which, on the one hand, connects crucibles with other items of the material culture and, on the other hand, bridges the gap between the consideration of the crucible as a medium for the transformation of matter, and its use as a vehicle for communication. The study of

this stamp may also serve as a starting point to address the relationships between metallurgy, chymistry and pottery making in the early modern world, and the links between these and other arenas of activity. Complementary lines of work may be the investigation of the relationship between crucible making and other ceramic technologies such as stoneware, and of the channels for the transport and trade of these vessels.

Finally, the focus on specific archaeological case studies of crucible use, considering the associated equipment and the specific reactions conducted, may allow a better insight into why and how different crucibles were used in each context. If specific patterns are found in the consumption and utilisation of technical ceramics, it may be possible to investigate whether ideas and skills were shared by those who had tools in common.

11.4. The analytical study of technical ceramics

This thesis adds to a growing body of analytical studies of technical ceramics used for metallurgical and chemical operations in archaeological contexts. The role of technical ceramics is increasingly recognised and investigated. In order to bear consistency, future research on chymistry and crucibles shall ideally follow a comparable analytical methodology, which has proved of use here as well as in previous work. Yet, it has been possible to identify some specific arenas for original analytical research which will be of aid in the technical interpretation of this type of ceramics.

Firstly, the characterisation of crucibles should pay attention not only to the thermal refractoriness, but also to the chemical refractoriness and corrosion resistance of the fabrics, which may be related to the composition of the paste, its vitrification state after prefiring and the surface finish of the vessel. This seems to have been a key factor behind the especially satisfactory performance of the more famous crucible productions, more important than the resistance to high temperatures. Since resistance to corrosion was a function of the processes to be conducted within the vessel, it must be assessed in relation to these processes, rather than as an absolute measure. In the case of technical ceramics used to process hot lead oxide, it should be considered that relatively large quartz inclusions within the fabric may have served as a buffer to corrosion, in spite of the aggressiveness of lead oxide to silica, due to the slowness of the reaction with the relatively coarse grains.

Secondly, the effect of graphite inclusions in the material properties of ceramic fabrics remains to be addressed in more detail. Of particular interest are the oxidation behaviour of graphite and the changing performance of ceramics as graphite burns out. More generally, it would be worthwhile conducting experiments to assess whether the material properties of fabrics containing different aplastics vary with their repeated use at very high temperatures, often above those of the original prefiring.

Thirdly, when assessing the thermal refractoriness of ceramic matrices, the iron content as detected by chemical analyses may not be taken as a straightforward proxy of heat resistance. The redox atmosphere of utilisation will result in this iron being present within the fabric either as ferric oxide (Fe_2O_3), ferrous oxide (FeO) or metal, all three components with distinct chemical behaviour, thus affecting refractoriness in appreciably different ways.

Finally, the pilot study of the ash-based cupels opens a promising field for further work on technical ceramics. It may be the time to exploit the informative potential of these artefacts, so abundant in many archaeological assemblages, beyond their mere use as diagnostic remains of cupellation. Historical and archaeological information show a relatively wide variability in the manufacturing techniques, whose technical and social implications are yet to be clarified.

For these analytical developments to become meaningful within research agendas, they will necessitate an articulate set of interpretative categories and concepts, as stressed throughout this thesis.

11.5. Theory and archaeometry

At the specific level of artefact analysis, it is useful to bear in mind the differences among formal properties, material properties and performance characteristics of the objects. Even if these categories often overlap, they provide a sound basis for the systematic characterisation and interpretation of technological systems. The concept of performance challenges the validity of general comparisons of the properties of artefacts in modern technical terms, while in turn it fosters a more socially informed comprehension of the production and consumption of artefacts. Instead of asking 'what did they do in order to make their crucibles refractory?', we may wish to ask 'why did they use graphitic clays?'.

This thesis has advocated the understanding of performance in the widest sense. When discussing the performance of a Renaissance crucible, we need to pay attention to its specific conditions of utilisation in the laboratory, and how it serves a given purpose in the furnace; but the crucible will also have performed a particular role in the market, conditioning its purchase by the user, and its acquisition and utilisation inevitably convey a series of social, cultural and economic connotations. If we try to split these as related, for example, to either utilitarian or social roles, we will be imposing an artificial hierarchy of primary and secondary applications. Similarly, trying to interpret the features of an artefact as either functional or stylistic entails judging what is essential and what accessory, and this assessment will usually be based on modern concepts that differ from past ones.

Also the concept of technological choice has been used in a broad sense, to incorporate the discussion of decisions made in the manufacture as well as in the attainment and use of the artefacts. The identification and interpretation of choices offers a useful strategy to understand the human agents in past technologies. By combining scientific analysis, historical research and anthropological approaches to material culture, it was possible to show that perception played a crucial role in decision making. It follows that the sensorial materiality of the artefact should be given a central place in the study of material culture, even when aided by scientific analyses of its invisible dimensions. From this perspective, the artefact emerges as an active constituent of the sociotechnical system rather than as a passive tool to be objectively understood. This realisation bridges a link between archaeology and modern laboratory studies, and underpins the focus on the material instrument taken up in this thesis.

In addition, the approach to the decisions made around the production and consumption of crucibles put on view the fact that the reasons behind a choice would not always be articulated or verbalised. The combination of all the available sources of information becomes thus mandatory, if we are to reconstruct a history that is not constrained to what people wrote about themselves.

11.6. Science, archaeology and history

An underlying theme of this thesis is the pertinence of using archaeological information in the study of historical periods and, more precisely, how scientific archaeology may fit in this panorama. Documentary sources were written with specific

purposes, and these should be investigated before their content is taken at face value. Furthermore, the aims of books often differ from the aims of human actions leaving material remains other than written sources, hence historical and archaeological information are complementary rather than redundant. Not every aspect of past societies was written about and, even when a topic is addressed in books, written information may not be easy to decipher, as seen in the case of some chymical sources. The study of material remains of historical societies offers an avenue to the nonverbal dimensions of human behaviour, and archaeological science, insofar as it helps exploit the informative potential of materials, further strengthens the role of archaeological research. Some findings of this thesis may serve to exemplify the successful combination of science, archaeology and history.

Archaeological research led to question the original assumption, based on written information, that the crucibles used in Oberstockstall had been produced in Obernzell. The stamp displayed on some black crucibles and other artefacts, related to masonic symbology, could only be comprehended by resorting to a wide range of sources of different types. The manufacture of the cupels within the laboratory matched historical accounts, but full analytical investigation of archaeological samples revealed hitherto unrecorded practices such as the recycling of raw materials. At a different scale, the study of archaeological remains from historical contexts illustrates the intricate nature of transition periods, for example showing the laboratory as a new creation of the Renaissance, but also as a result of long-term developments engrained in medieval and earlier technological practice. Similarly, only the integration of scientific, archaeological and historical data could provide a balanced picture of the production of crucibles, including home made, moulded, usually grog-tempered vessels, as well as sand-tempered and graphitic ones, wheel-thrown and manufactured industrially in two different centres.

All in all, a theoretically informed interdisciplinary research programme has proved the most effective approach to the complexities of Renaissance technology and science. In this light, the questions to be addressed by those studying the past need not to be constrained to the realm of 'what did they do?' but may expand onto aspects such as 'why did they do what they did?' or even 'why did they write what they wrote about what they did?' Much more work is yet to be done but, hopefully, this thesis has offered a way to go forward.

11.7. The archaeology of science and technology

Perhaps one of the milestones to remain more prominently in the aftermath of this work is the newly defined archaeology of alchemy and chemistry. By focussing on the material remains from the sacristy in Oberstockstall, an archaeological approach avoided unquestioned assumptions and started by addressing the practical side of human activities. This led to independent information about the trade and supply of specialised pieces of equipment, their technical properties, specific uses and performance, hence offering a privileged view to the emergence of the scientific laboratory. Besides, the archaeological remains illustrate the transfer of utensils and skills among various realms of early science and industry, whilst allowing the identification of specific choices in raw materials and reactions. Finally, perhaps most importantly, archaeology reveals information regarding the perception and understanding of different materials by past peoples. The ultimate aim of Renaissance chymistry was the understanding and manipulation of natural elements, and now, studying how different materials were selected and used, we can effectively assess how these practical activities may have influenced the ways in which their nature and properties were perceived.

Just as Renaissance chymists found difficulties in understanding the materials they handled and processed, so do we face the challenge of coming to terms with the wealth of materials they left behind. Even so, the archaeology of alchemy and chemistry is emerging as a fruitful complementary approach to the history of science and technology. The successful combination of archaeological and historical sources requires defying traditional constraints and encouraging systematic co-operation between scientists, archaeologists and historians, yet it promises a very exciting future. It is eagerly hoped that this thesis shall serve as an inspiration for further research in this field.

Appendix 1. Chemical composition of ceramic matrices

1. Crucibles from Oberstockstall

Table 20. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of crucibles from Oberstockstall.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO	ZnO	Sb_2O_3	PbO
OB 286	**0.5**	**1.1**	**28.5**	**55.6**	**0.2**	**3.0**	-	**2.3**	**3.0**	**1.4**	**4.5**	-	-	-
Std. dev.	0.32	0.61	1.73	1.97	0.14	1.81	-	0.55	1.07	0.33	0.54	-	-	-
Max.	1.2	2.5	30.7	58.3	0.4	6.0	-	3.2	4.7	2.0	5.5	-	-	-
Min.	0.2	0.8	25.7	53.7	0.0	0.8	-	1.6	1.6	1.0	3.8	-	-	-
OB 288	**0.1**	**0.6**	**33.1**	**58.7**	**0.4**	-	-	**1.5**	**0.6**	**1.1**	**3.9**	-	-	-
Std. dev.	0.07	0.09	0.34	0.34	0.12	-	-	0.14	0.08	0.06	0.38	-	-	-
Max.	0.2	0.7	33.6	59.2	0.5	-	-	1.6	0.7	1.2	4.3	-	-	-
Min.	0.0	0.5	32.9	58.5	0.3	-	-	1.3	0.5	1.1	3.6	-	-	-
OB 290	**0.2**	**0.7**	**32.2**	**57.5**	**0.2**	-	-	**1.9**	**0.6**	**1.2**	**5.3**	-	-	-
Std. dev.	0.06	0.21	0.92	0.77	0.11	-	-	0.29	0.16	0.09	0.34	-	-	-
Max.	0.2	1.0	33.5	58.3	0.3	-	-	2.2	0.9	1.3	5.8	-	-	-
Min.	0.1	0.5	31.2	56.3	0.1	-	-	1.5	0.5	1.1	5.0	-	-	-
OB 307	**2.5**	**0.8**	**31.2**	**55.0**	**0.2**	-	**0.7**	**5.5**	**0.6**	**1.2**	**2.4**	-	-	-
Std. dev.	0.54	0.13	0.40	1.09	0.03	-	0.03	0.34	0.18	0.18	0.14	-	-	-
Max.	3.3	0.9	31.8	56.0	0.2	-	0.7	5.9	0.9	1.3	2.6	-	-	-
Min.	2.1	0.6	30.8	54.0	0.2	-	0.6	5.1	0.4	1.0	2.2	-	-	-
OB 345	**0.4**	**0.5**	**30.4**	**57.4**	**0.3**	**3.3**	-	**2.1**	**0.6**	**1.3**	**3.6**	-	-	-
Std. dev.	0.06	0.13	1.62	2.63	0.19	1.35	-	0.08	0.07	0.15	0.84	-	-	-
Max.	0.4	0.7	31.5	59.4	0.4	4.8	-	2.2	0.7	1.4	4.5	-	-	-
Min.	0.3	0.4	28.5	54.4	0.1	2.2	-	2.0	0.5	1.1	2.8	-	-	-
OB 394	**0.1**	**0.8**	**30.9**	**58.5**	**0.2**	-	-	**2.3**	**0.6**	**1.3**	**5.3**	-	-	-
Std. dev.	0.14	0.14	1.08	1.75	0.28	-	-	0.27	0.15	0.40	0.56	-	-	-
Max.	0.3	1.0	32.3	61.1	0.6	-	-	2.7	0.8	1.8	6.0	-	-	-
Min.	0.0	0.5	29.1	55.8	0.0	-	-	1.9	0.4	0.7	4.2	-	-	-
OB 395	**0.3**	**0.7**	**33.4**	**56.2**	**0.3**	-	**0.2**	**2.1**	**0.6**	**1.3**	**5.1**	-	-	-
Std. dev.	0.12	0.30	1.23	0.94	0.17	-	0.13	0.23	0.22	0.22	0.46	-	-	-
Max.	0.5	1.2	35.2	57.4	0.4	-	0.3	2.4	0.8	1.7	6.0	-	-	-
Min.	0.1	0.4	32.3	55.2	0.0	-	0.0	1.9	0.3	1.1	4.7	-	-	-
OB 466	**0.5**	**0.5**	**32**	**56.3**	**0.2**	**0.2**	**≤0.1**	**2.5**	**0.7**	**1.4**	**5.6**	-	-	-
Std. dev.	0.06	0.10	1.08	0.68	0.09	0.09	0.07	0.31	0.23	0.31	0.59	-	-	-
Max.	0.6	0.6	33.5	57.0	0.3	0.3	0.2	3.0	1.0	1.9	6.7	-	-	-
Min.	0.4	0.4	30.5	55.1	0.1	0.1	0.0	2.1	0.5	1.0	4.9	-	-	-

Table 20 (cont.)

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO	ZnO	Sb_2O_3	PbO
OB 494	**2.5**	**1.2**	**29.3**	**55.4**	**0.3**	-	**1.2**	**7.2**	**0.6**	**1.2**	**1.3**	-	-	-
Std. dev.	0.11	0.30	3.56	3.95	0.13	-	0.65	0.29	0.05	0.15	0.18	-	-	-
Max.	2.6	1.5	32.5	59.6	0.5	-	1.9	7.4	0.6	1.3	1.4	-	-	-
Min.	2.4	0.9	25.4	51.8	0.2	-	0.6	6.9	0.6	1.0	1.1	-	-	-
OB 495	**0.2**	**0.4**	**32.7**	**53.9**	**0.4**	-	-	**1.8**	**0.6**	**1.4**	**7.9**	-	-	**0.7**
Std. dev.	0.07	0.16	2.04	2.49	0.19	-	-	0.18	0.05	0.29	3.45	-	-	0.42
Max.	0.3	0.6	34.6	56.2	0.6	-	-	1.9	0.7	1.7	11.9	-	-	1.2
Min.	0.2	0.3	30.5	51.3	0.2	-	-	1.6	0.6	1.1	5.8	-	-	0.3
OB 515	**0.5**	**0.7**	**34.0**	**57.3**	**0.5**	**0.2**	**0.2**	**2.5**	**0.5**	**1.4**	**2.5**	-	-	-
Std. dev.	0.07	0.09	1.92	1.56	0.15	0.17	0.06	0.08	0.14	0.16	0.95	-	-	-
Max.	0.6	0.8	36.5	59.2	0.7	0.2	0.3	2.6	0.6	1.5	3.9	-	-	-
Min.	0.4	0.5	32.4	55.4	0.4	0.0	0.2	2.5	0.4	1.1	1.7	-	-	-
OB 519F	**0.3**	**0.7**	**33.9**	**56.2**	**0.4**	-	-	**2.0**	**1.0**	**1.1**	**4.5**	-	-	-
Std. dev.	0.11	0.41	1.39	1.60	0.06	-	-	0.37	0.27	0.26	0.32	-	-	-
Max.	0.4	1.5	34.8	59.4	0.5	-	-	2.7	1.4	1.4	5.1	-	-	-
Min.	0.1	0.4	31.1	55.0	0.3	-	-	1.6	0.7	0.7	4.1	-	-	-
OB 520	**1.9**	**0.7**	**32.8**	**54.7**	**0.4**	-	**0.8**	**2.8**	**0.4**	**1.3**	**4.0**	-	-	-
Std. dev.	0.71	0.11	0.87	0.51	0.16	-	0.3	0.35	0.10	0.42	0.86	-	-	-
Max.	2.6	0.9	33.7	55.1	0.5	-	1.3	3.1	0.5	1.9	5.2	-	-	-
Min	1.0	0.6	32.0	54.0	0.2	-	0.6	2.3	0.2	1.0	3.2	-	-	-
OB 560	**0.2**	**0.7**	**30.8**	**56.9**	**0.2**	-	-	**2.8**	**0.8**	**1.6**	**6.1**	-	-	-
Std. dev.	0.14	0.14	1.07	1.78	0.33	-	-	0.64	0.20	0.30	0.63	-	-	-
Max.	0.4	0.8	32.2	58.8	0.5	-	-	3.6	1.0	2.1	7.2	-	-	-
Min.	0.0	0.5	29.2	54.0	0.0	-	-	1.8	0.6	1.2	5.5	-	-	-
OB 569	**0.3**	**0.5**	**34.4**	**56**	**0.3**	**0.6**	**≤0.1**	**1.5**	**1.6**	**1.5**	**3.3**	-	-	-
Std. dev.	0.04	0.06	0.35	0.67	0.10	0.17	0.02	0.19	0.27	0.13	0.12	-	-	-
Max.	0.3	0.6	34.9	56.7	0.4	0.9	0.1	1.8	2.0	1.6	3.4	-	-	-
Min.	0.2	0.5	33.9	54.9	0.2	0.5	0.0	1.4	1.3	1.3	3.2	-	-	-
OB n001	**0.4**	**0.8**	**30.1**	**55.1**	**0.2**	-	-	**2.5**	**1.2**	**1.5**	**7.1**	**0.2**	**1.0**	-
Std. dev.	0.08	0.2	1.26	1.73	0.17	-	-	0.43	0.36	0.23	1.18	0.28	0.15	-
Max.	0.5	1.0	31.9	57.1	0.3	-	-	3.1	1.7	1.9	8.2	0.5	1.2	-
Min.	0.3	0.5	28.5	53.4	0.0	-	-	2.1	0.9	1.3	5.2	0.0	0.9	-

2. Scorifiers from Oberstockstall

Table 21. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of scorifiers from Oberstockstall.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO	As_2O_3	SnO_2
OB 268	**0.2**	**0.7**	**33.2**	**58.9**	**0.3**	-	-	**1.8**	**0.6**	**0.6**	**3.6**	-	-
Std. dev.	0.05	0.02	0.68	0.85	0.21	-	-	0.38	0.06	0.15	0.17	-	-
Max.	0.3	0.7	33.9	59.6	0.7	-	-	2.3	0.7	0.9	3.8	-	-
Min.	0.2	0.6	32.3	57.9	0.2	-	-	1.4	0.5	0.5	3.4	-	-
OB 269	**0.4**	**1.0**	**26.0**	**62.8**	**0.3**	-	-	**1.8**	**2.2**	**0.8**	**4.3**	**0.2**	**0.3**
Std. dev.	0.25	0.20	1.73	2.80	0.13	-	-	0.23	0.78	0.09	0.27	0.23	0.12
Max.	0.9	1.3	28.4	66.3	0.4	-	-	2.2	3.4	0.9	4.7	0.4	0.4
Min.	0.3	0.8	23.7	59.0	0.1	-	-	1.6	1.4	0.7	4.1	0.0	0.1
OB 276	**0.3**	**0.4**	**31.4**	**59.6**	**0.1**	-	**0.2**	**1.9**	**0.7**	**0.7**	**4.8**	-	-
Std. dev.	0.05	0.08	1.35	1.38	0.15	-	0.06	0.10	0.16	0.06	0.26	-	-
Max.	0.3	0.5	33.3	60.6	0.2	-	0.3	2.0	0.9	0.8	5.1	-	-
Min.	0.2	0.3	30.2	57.6	0.0	-	0.1	1.8	0.6	0.6	4.5	-	-
OB 277	**0.2**	**0.5**	**32.3**	**59.2**	**0.2**	-	-	**1.6**	**0.7**	**0.7**	**4.5**	-	-
Std. dev.	0.13	0.06	1.40	1.64	0.10	-	-	0.20	0.15	0.10	0.13	-	-
Max.	0.3	0.6	34.0	61.3	0.3	-	-	1.9	0.9	0.8	4.6	-	-
Min.	0.1	0.4	30.6	57.4	0.1	-	-	1.4	0.6	0.7	4.3	-	-
OB 279	**0.3**	**0.7**	**30.5**	**56.8**	**0.3**	-	-	**2.0**	**0.8**	**1.5**	**7.1**	-	-
Std. dev.	0.18	0.11	1.19	1.20	0.09	-	-	0.12	0.10	0.21	0.31	-	-
Max.	0.5	0.9	31.4	58.2	0.4	-	-	2.1	0.9	1.7	7.5	-	-
Min.	0.1	0.6	28.8	55.4	0.2	-	-	1.8	0.7	1.2	6.8	-	-
OB 281	**0.2**	**0.4**	**32.0**	**59.7**	**0.3**	-	-	**1.9**	**0.6**	**0.5**	**4.6**	-	-
Std. dev.	0.15	0.07	1.59	2.03	0.12	-	-	0.03	0.21	0.18	0.42	-	-
Max.	0.3	0.5	33.1	61.9	0.4	-	-	1.9	0.8	0.7	4.9	-	-
Min.	0.1	0.3	30.2	58.1	0.1	-	-	1.9	0.4	0.3	4.1	-	-
OB 282	**0.3**	**0.5**	**30.1**	**61.6**	**0.3**	-	-	**2.1**	**0.6**	**0.6**	**4.1**	-	-
Std. dev.	0.05	0.11	0.62	0.72	0.11	-	-	0.41	0.20	0.07	0.11	-	-
Max.	0.3	0.6	30.9	62.4	0.5	-	-	2.6	0.7	0.7	4.3	-	-
Min.	0.2	0.4	29.5	60.6	0.2	-	-	1.7	0.3	0.6	4.0	-	-
OB 284	**1.0**	**0.4**	**33.4**	**55.7**	**0.3**	-	-	**2.0**	**0.5**	**1.3**	**5.4**	-	-
Std. dev.	0.38	0.12	1.12	0.39	0.25	-	-	0.28	0.15	0.38	0.54	-	-
Max.	1.3	0.6	34.3	56.1	0.6	-	-	2.4	0.7	1.8	5.9	-	-
Min.	0.4	0.3	31.6	55.2	0.1	-	-	1.7	0.3	0.9	4.6	-	-
OB n006	**1.0**	**0.3**	**31.7**	**53.4**	**0.2**	**0.5**	**0.1**	**5.8**	**0.5**	**1.1**	**3.8**	**1.7**	-
Std. dev.	0.38	0.15	1.18	1.12	0.12	0.12	0.08	1.08	0.17	0.22	0.70	1.23	-
Max.	1.5	0.5	33.3	55.1	0.3	0.6	0.2	7.4	0.7	1.4	5.0	3.6	-
Min.	0.5	0.0	30.1	51.9	0.0	0.2	0.0	4.4	0.2	0.8	3.2	0.3	-

3. Cupels from Oberstockstall

Table 22. Chemical composition by SEM-EDS, normalised to 100 wt%, of the white powder interpreted as raw material for making cupels (OB 925), the unused cupel (OB 937), and the top layer of the latter (OB 937*), all from Oberstockstall.

	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	MnO	FeO	CuO	ZnO	Ag	PbO
OB 925	**1.9**	**3.3**	**11.8**	**26.4**	**3.7**	**0.4**	**1.1**	**44.8**	**-**	**4.6**	**2.2**	**-**	**≤0.2**	**-**
Std. dev.	0.81	1.04	2.91	2.01	0.77	0.29	0.37	2.52	-	1.54	1.35	-	0.06	-
Max.	3.2	5.5	17.9	28.9	5.5	0.7	1.9	47.8	-	8.1	4.5	-	0.2	-
Min.	0.0	1.8	7.1	21.5	2.5	0.0	0.6	38.6	-	3.3	0.0	-	0.0	-
OB 937	**3.5**	**2.3**	**13.5**	**12.1**	**0.3**		**1.3**	**64.6**	**-**	**1.8**	**0.9**	**0.1**	**-**	**0.7**
Std. dev.	0.59	0.53	3.06	6.42	0.57	-	0.41	2.31	-	0.48	0.35	0.24	-	0.34
Max.	4.3	2.8	16.0	26.1	1.2	-	1.8	67.5	-	2.3	1.3	0.5	-	1.3
Min.	2.3	1.3	7.1	8.1	0.0	-	0.5	61.1	-	0.8	0.2	0.0	-	0.2
OB 937*	**5.0**	**3.3**	**18.6**	**9.7**	**0.3**		**1.3**	**59.7**	**0.3**	**1.8**	**-**	**-**	**-**	**-**
Std. dev.	1.79	0.91	3.23	2.88	0.48	-	0.25	8.21	0.48	0.63	-	-	-	-
Max.	6.5	4.2	21.4	13.5	1.1	-	1.6	75.6	1.0	2.9	-	-	-	-
Min.	1.6	1.6	12.6	6.2	0.0	-	1.0	53.8	0.0	1.4	-	-	-	-

Table 23. Chemical composition by SEM-EDS, normalised to 100 wt%, of the larger crystals identified within the white powder interpreted as raw material for making cupels (OB 925) and the fabric of the unused cupel (OB 937) from Oberstockstall.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	MnO	FeO	CuO	As_2O_3	Ag	PbO
OB 925 1	-	-	2.1	5.0	11.0	25.6	-	-	52.4	-	-	3.9	-	-	-	-
2	-	0.2	0.4	1.4	14.6	1.1	0.3	0.2	78.8	-	-	1.4	1.6	-	-	-
3	-	1.2	1.2	8.5	27.7	2.2	0.9	0.5	54.4	-	-	3.2	0.5	-	-	-
4	-	2.0	1.9	8.2	31.5	1.3	0.8	0.6	48.8	-	-	3.9	1.7	-	-	-
5	-	1.9	5.4	31.7	15.8	8.7	0.4	1.4	28.9	-	-	2.6	2.6	1.0	-	-
6	-	2.1	3.2	26.6	25.7	1.3	-	2.1	15.5	-	-	18.9	2.2	2.6	-	-
7	-	2.8	1.4	4.8	37.9	1.6	-	0.3	49.2	-	-	1.0	0.9	-	-	-
8	-	0.8	0.8	1.3	36.5	0.7	0.4	-	58.5	-	-	-	1.1	-	-	-
9	-	1.2	8.4	34.4	17.8	1.5	-	1.0	33.4	-	-	2.2	-	-	-	
10	-	-	-	4.5	14.0	-	-	-	68.0	-	-	13.4	-	-	-	
11	-	2.1	-	5.3	40.1	-	-	0.5	49.0	-	-	1.2	-	1.9	-	-
12	-	1.3	3.5	8.2	35.6	-	-	0.6	48.2	-	-	2.8	-	-	-	-
13	-	1.2	-	42.7	27.2	-	-	-	28.9	-	-	-	-	-	-	-
14	-	1.8	4.7	46.0	17.7	2.3	-	1.3	22.9	-	-	3.3	-	-	-	-
OB 937 1	0.4	1.2	0.4	2.1	37.0	-	0.1	-	56.6	-	-	0.4	-	-	0.6	-
2	-	0.9	0.4	2.4	28.2	0.6	-	-	66.5	-	-	-	-	-	-	1.0
3	-	1.6	0.5	4.1	32.8	-	0.4	-	60.0	-	-	0.6	-	-	-	-
4	0.3	3.7	3.0	15.5	8.2	0.6	-	1.8	62.9	-	-	3.3	-	-	-	0.9
5	-	0.3	0.5	4.1	1.4	0.7	-	0.8	90.0	-	-	2.3	-	-	-	-
6	-	2.2	1.1	7.6	4.7	0.7	-	0.6	82.2	-	-	1.0	-	-	-	-
7	0.7	4.2	10.0	24.5	3.0	0.6	-	9.2	35.9	2.0	-	8.9	-	-	-	-
8	-	7.8	6.5	40.2	6.9	0.5	-	1.4	30.6	-	-	5.0	1.0	-	-	-
9	-	2.0	1.3	8.3	7.0	0.7	-	0.7	77.8	-	0.4	0.9	0.8	-	-	-
10	-	3.6	3.0	15.8	7.9	-	-	1.7	66.1	-	-	1.1	-	-	-	-

Table 24. Chemical composition by SEM-EDS, normalised to 100 wt%, of submetallic particles identified within the white powder interpreted as raw material for making cupels (OB 925) and the fabric of the unused cupel (OB 937) from Oberstockstall.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	MnO	FeO	NiO	CuO	ZnO	As_2O_3	Br	Ag	SnO_2	Sb_2O_3	PbO
OB 925 1	-	0.9	1.8	11.6	6.6	-	2.9	0.3	11.0	-	-	1.2	-	54.5	3.8	1.5	-	-	-	-	3.9
2	-	2.1	-	5.0	7.8	16.2	-	0.4	9.2	-	0.6	53.6	-	2.3	-	0.7	1.3	0.9	-	-	-
3	-	0.9	0.6	3.6	10.8	1.2	0.4	-	14.0	-	-	4.8	1.3	46.7	6.4	0.8	-	-	7.5	-	1.2
4	-	1.6	1.5	4.1	6.0	1.2	-	-	7.0	-	-	1.0	-	1.0	-	-	-	-	76.7	-	-
5	-	0.9	-	2.4	13.5	1.1	-	-	16.4	-	-	0.9	-	63.9	-	-	1.0	-	-	-	-
6	-	-	-	-	4.2	-	-	-	26.2	-	-	48.2	-	14.9	-	0.2	-	-	-	-	6.4
7	-	-	1.3	2.8	6.2	-	2.2	1.4	47.3	-	-	3.6	-	13.7	-	-	-	-	-	-	21.5
8	-	3.0	1.1	3.3	4.7	-	3.1	-	9.4	-	0.8	45.0	-	16.2	13.6	-	-	-	-	-	-
9	-	-	-	4.3	14.4	-	1.6	-	25.5	-	-	0.8	-	-	-	13.6	1.5	-	-	-	38.5
10	-	-	2.5	10.4	5.9	3.1	9.8	-	9.3	-	-	1.8	-	37.1	3.6	6.5	-	-	4.4	-	5.6
11	-	-	3.7	7.4	22.3	1.5	2.4	0.8	32.3	-	-	0.8	-	5.4	-	9.4	-	-	-	-	14.1
12	-	-	1.1	5.4	19.7	1.4	1.6	-	40.3	-	-	2.1	-	16.8	-	3.3	-	-	-	-	8.3
13	-	-	-	5.7	32.4	2.1	0.6	0.5	43.8	-	-	2.4	-	1.0	-	3.4	1.4	1.1	-	-	5.6
14	-	1.4	2.3	13.2	25.4	1.2	0.7	1.0	51.7	-	-	3.1	-	-	-	-	-	-	-	-	-
15	-	2.1	3.0	6.7	24.6	1.6	1.8	-	31.5	-	-	1.0	-	27.6	-	-	-	-	-	-	-
OB 937 1	0.3	1.1	0.7	3.6	1.9	-	-	-	25.4	-	-	0.3	-	0.6	-	-	-	-	-	61.1	5.1
2	-	4.6	1.6	12.3	3.0	-	0.5	1.8	3.6	2.5	-	45.7	-	2.0	-	-	-	-	4.4	-	17.8
3	-	-	1.5	61.8	8.9	-	0.3	1.0	16.5	-	-	1.4	-	-	-	2.5	-	-	-	-	6.1
4	-	0.9	-	2.4	1.0	-	-	-	5.4	-	-	5.0	-	-	-	-	-	-	85.3	-	-
5	-	1.6	0.7	4.7	1.9	-	-	-	30.8	-	-	0.7	-	-	-	-	-	-	-	59.7	-
6	-	4.9	3.6	9.2	1.4	-	-	-	26.8	-	-	3.0	-	1.0	-	-	-	-	-	48.9	1.2

4. Comparative material from Oberstockstall

Table 25. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of artefacts from the Oberstockstall assemblage used as comparative material.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO
OB 17 (capital)	**0.3**	**0.7**	**26.0**	**65.6**	**0.2**	-	-	**1.7**	**1.1**	**1.4**	**2.9**
Std. dev.	0.13	0.06	1.21	1.74	0.04	-	-	0.22	0.31	0.20	0.18
Max.	0.5	0.8	27.5	68.3	0.3	-	-	2.0	1.6	1.8	3.1
Min.	0.1	0.6	24.5	64.1	0.2	-	-	1.5	0.8	1.3	2.7
OB 362 (stamped)	**0.2**	**1.0**	**27.1**	**59.0**	**0.2**	-	-	**1.7**	**1.0**	**1.4**	**8.3**
Std. dev.	0.10	0.21	0.70	1.03	0.11	-	-	0.17	0.33	0.13	0.43
Max.	0.4	1.2	27.6	60.0	0.4	-	-	1.9	1.6	1.6	8.8
Min.	0.1	0.7	25.9	57.3	0.1	-	-	1.5	0.8	1.2	7.9
OB 389 (unstamped)	**0.3**	**0.9**	**31.8**	**55.0**	**0.2**	**1.3**	-	**1.7**	**3.7**	**0.7**	**4.4**
Std. dev.	0.11	0.11	0.70	0.47	0.13	0.23	-	0.08	0.57	0.13	0.11
Max.	0.5	1.0	32.4	55.5	0.3	1.5	-	1.8	4.4	0.8	4.5
Min.	0.2	0.8	30.9	54.6	0.0	1.0	-	1.6	3.1	0.5	4.3
OB n002 (cucurbit)	**0.5**	**0.4**	**23.1**	**66.7**	**0.2**	-	-	**5.2**	**0.4**	**1.1**	**2.4**
Std. dev.	0.04	0.13	1.87	1.96	0.06	-	-	0.27	0.06	0.49	0.10
Max.	0.5	0.6	25.1	68.1	0.2	-	-	5.4	0.5	1.7	2.4
Min.	0.4	0.3	21.4	64.5	0.1	-	-	4.9	0.4	0.8	2.2
OB n003 (orange pot)	**0.2**	**0.5**	**31.8**	**59.5**	**0.2**	-	-	**1.2**	**1.8**	**0.7**	**4.2**
Std. dev.	0.06	0.02	1.09	1.22	0.10	-	-	0.16	0.31	0.24	0.11
Max.	0.2	0.5	33.0	60.3	0.3	-	-	1.4	2.2	0.8	4.3
Min.	0.1	0.5	31.1	58.1	0.2	-	-	1.1	1.6	0.4	4.1
OB n004 ('sandbath')	**0.2**	**0.7**	**32.0**	**56.1**	**0.2**	-	-	**1.9**	**1.1**	**1.6**	**6.4**
Std. dev.	0.08	0.11	1.71	2.04	0.15	-	-	0.20	0.55	0.39	0.22
Max.	0.3	0.9	34.6	59.3	0.4	-	-	2.1	1.9	2.3	6.7
Min.	0.1	0.6	29.8	54.3	0.0	-	-	1.5	0.6	1.2	6.1
OB n005 ('trumpet')	**0.3**	**0.9**	**20.9**	**69.6**	**0.1**	-	-	**2.1**	**1.6**	**1.6**	**2.9**
Std. dev.	0.20	0.25	2.53	2.98	0.10	-	-	0.18	0.47	0.27	0.15
Max.	0.7	1.3	23.7	73.9	0.2	-	-	2.3	2.4	1.9	3.1
Min.	0.2	0.7	17.5	66.3	0.0	-	-	1.9	1.2	1.2	2.7
lute OB 495 (crucible)	**0.5**	**4.6**	**11.8**	**44.0**	**0.9**	**0.6**	**0.8**	**0.8**	**24.3**	**1.0**	**10.8**
Std. dev.	0.17	1.55	1.04	10.38	0.28	0.26	0.46	0.06	4.30	1.22	6.21
Max.	0.6	6.0	12.7	55.9	1.1	0.9	1.3	0.8	29.2	2.4	17.7
Min.	0.3	3.0	10.7	37.9	0.6	0.4	0.4	0.7	21.1	0.2	5.7
lute OB n002 (cucurbit)	**0.7**	**3.6**	**9.2**	**51.6**	**0.4**	-	**0.4**	**4.2**	**25.5**	**0.3**	**4.0**
Std. dev.	0.28	1.29	1.24	11.31	0.01	-	0.00	0.83	7.13	0.11	0.61
Max.	0.9	4.6	10.1	59.6	0.4	-	0.4	4.8	30.6	0.4	4.5
Min.	0.5	2.7	8.4	43.6	0.4	-	0.4	3.6	20.5	0.3	3.6

5. Ceramic artefacts from Obernzell

Table 26. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of artefacts from Obernzell.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	K_2O	CaO	TiO_2	FeO
OBZL 01 (crucible)	**1.1**	**1.0**	**27.7**	**58.3**	**0.4**	**-**	**2.5**	**0.9**	**1.2**	**6.9**
Std. dev.	0.32	0.75	0.51	2.10	0.17	-	0.46	0.29	0.32	1.28
Max.	1.5	2.2	28.4	61.3	0.5	-	3.1	1.4	1.6	8.2
Min.	0.8	0.3	27.1	55.6	0.1	-	2.1	0.7	0.8	4.9
OBZL 02 (handle)	**0.4**	**0.7**	**26.6**	**55.3**	**4.9**	**0.8**	**1.3**	**3.0**	**1.2**	**5.9**
Std. dev.	0.11	0.18	1.05	1.45	1.21	0.99	0.10	0.20	0.20	0.38
Max.	0.5	1.0	28.3	57.1	6.3	2.4	1.5	3.2	1.5	6.3
Min.	0.2	0.4	25.2	53.7	3.6	0.0	1.2	2.7	0.9	5.3
OBZL 03 (lid)	**0.7**	**1.2**	**26.0**	**58.4**	**1.0**	**-**	**2.1**	**1.1**	**1.3**	**8.2**
Std. dev.	0.27	0.14	0.17	0.89	0.21	-	0.23	0.20	0.10	0.51
Max.	1.1	1.4	26.3	59.5	1.2	-	2.3	1.4	1.4	8.9
Min.	0.5	1.1	25.9	57.3	0.7	-	1.8	0.9	1.1	7.8
OBZL 04 (rim)	**0.3**	**0.7**	**28.2**	**55.1**	**0.6**	**-**	**1.6**	**1.4**	**1.6**	**10.5**
Std. dev.	0.22	0.10	0.57	1.14	0.39	-	0.15	0.18	0.20	0.88
Max.	0.6	0.8	28.8	56.2	1.1	-	1.8	1.6	1.9	11.8
Min.	0.1	0.6	27.4	53.5	0.3	-	1.4	1.2	1.4	10.0

6. Crucibles from the Old Ashmolean laboratory, Oxford

Table 27. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of crucibles from the Old Ashmolean laboratory.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
OX 1422	**0.3**	**0.8**	**28.9**	**55.5**	**0.2**	**2.6**	**0.9**	**1.4**	**9.3**
Std. dev.	0.13	0.24	1.11	1.04	0.11	0.41	0.13	0.16	0.60
Max.	0.5	1.1	29.9	57.1	0.4	3.1	1.0	1.6	9.9
Min.	0.1	0.4	26.8	54.1	0.1	2.0	0.7	1.2	8.2
OX 1413	**0.4**	**0.2**	**36.4**	**58.0**	**0.2**	**1.9**	**0.2**	**1.6**	**1.2**
Std. dev.	0.09	0.09	0.42	0.42	0.15	0.04	0.06	0.35	0.13
Max.	0.5	0.3	36.9	58.6	0.3	2.0	0.3	2.1	1.3
Min.	0.3	0.1	35.8	57.5	0.0	1.9	0.1	1.2	1.0
OX n001	**0.2**	**0.5**	**36.5**	**57.1**	**0.2**	**1.4**	**0.2**	**2.0**	**2.0**
Std. dev.	0.04	0.07	0.47	0.52	0.14	0.08	0.10	0.18	0.11
Max.	0.2	0.6	37.0	58.0	0.4	1.5	0.4	2.2	2.1
Min.	0.2	0.5	36.0	56.6	0.1	1.3	0.1	1.7	1.9

7. Crucibles from Großalmerode

Table 28. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of crucibles from Großalmerode.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
Groß 52205Z	**0.1**	**0.5**	**37.5**	**56.5**	**0.4**	**1.2**	**0.3**	**1.9**	**1.7**
Std. dev.	0.10	0.10	0.46	0.43	0.14	0.16	0.05	0.04	0.09
Max.	0.2	0.7	38.2	57.1	0.6	1.4	0.4	1.9	1.9
Min.	0.0	0.4	37.0	55.8	0.2	1.0	0.2	1.8	1.6
Groß 52207V	**0.1**	**0.5**	**36.9**	**56.8**	**0.2**	**1.5**	**0.3**	**1.9**	**1.9**
Std. dev.	0.09	0.12	0.21	0.24	0.11	0.51	0.08	0.12	0.15
Max.	0.2	0.6	37.2	57.1	0.4	2.4	0.4	2.1	2.1
Min.	0.0	0.3	36.7	56.5	0.2	1.2	0.2	1.8	1.7

8. Crucibles from the Cripplegate Buildings, London

Table 29. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of crucibles from the Cripplegate Buildings.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO	ZnO
WFG 118/107	**0.2**	**0.2**	**17.8**	**77.3**	**0.1**	**1.2**	**0.4**	**1.2**	**1.0**	**0.7**
Std. dev.	0.14	0.09	1.91	2.47	0.13	0.19	0.08	0.15	0.12	0.54
Max.	0.4	0.3	21.2	79.6	0.2	1.5	0.5	1.5	1.1	1.5
Min.	0.0	0.1	15.8	73.4	0.0	1.0	0.3	1.1	0.8	0.1
WFG 118/114	**0.7**	**0.8**	**23.9**	**68.5**	**0.3**	**2.1**	**0.7**	**1.3**	**1.7**	**-**
Std. dev.	0.16	0.12	1.08	1.88	0.17	0.13	0.08	0.21	0.20	-
Max.	0.9	1.0	24.9	71.7	0.5	2.2	0.8	1.6	1.9	-
Min.	0.5	0.7	22.1	66.9	0.0	1.9	0.6	1.0	1.5	-
WFG 18/116	**0.5**	**0.5**	**36.6**	**57.0**	**0.3**	**1.2**	**0.2**	**1.8**	**2.0**	**-**
Std. dev.	0.15	0.05	0.38	0.50	0.05	0.29	0.08	0.07	0.31	-
Max.	0.6	0.5	37.2	57.6	0.4	1.7	0.3	1.9	2.4	-
Min.	0.3	0.4	36.3	56.4	0.3	1.0	0.1	1.7	1.7	-
WFG 118/121	**0.3**	**1.2**	**22.6**	**66.1**	**0.1**	**4.3**	**0.5**	**1.0**	**3.8**	**-**
Std. dev.	0.13	0.08	1.39	1.56	0.11	0.39	0.04	0.34	0.42	-
Max.	0.5	1.3	24.2	68.2	0.2	4.7	0.6	1.4	4.3	-
Min.	0.2	1.1	20.9	64.4	0.0	4.0	0.5	0.6	3.3	-
WFG 18/129	**0.1**	**0.5**	**36.9**	**56.8**	**0.3**	**1.2**	**0.2**	**1.8**	**2.2**	**-**
Std. dev.	0.04	0.08	0.40	0.34	0.12	0.23	0.11	0.07	0.25	-
Max.	0.2	0.6	37.4	57.3	0.4	1.5	0.3	1.9	2.6	-
Min.	0.1	0.4	36.3	56.3	0.1	0.9	0.0	1.7	1.9	-

9. Crucibles from Jamestown

Table 30. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of crucibles from Jamestown.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
A631/CC2	**0.1**	**0.5**	**35.6**	**57.8**	**0.4**	**1.2**	**0.4**	**2.2**	**1.7**
Std. dev.	0.03	0.03	0.68	0.76	0.03	0.14	0.05	0.11	0.08
Max.	0.2	0.6	36.0	58.9	0.4	1.4	0.5	2.3	1.8
Min.	0.1	0.5	34.6	57.2	0.3	1.1	0.4	2.1	1.7
JR 1024/CC3	**0.2**	**0.4**	**36.8**	**56.4**	**0.3**	**1.5**	**0.2**	**1.9**	**2.3**
Std. dev.	0.04	0.14	0.28	0.43	0.08	0.51	0.04	0.06	0.77
Max.	0.2	0.5	37.0	56.9	0.4	1.9	0.2	1.9	3.2
Min.	0.1	0.3	36.5	56.1	0.3	0.9	0.2	1.8	1.8
JR 124F/C1	**0.2**	**0.5**	**36.8**	**57.1**	**0.2**	**1.1**	**0.4**	**2.0**	**1.8**
Std. dev.	0.05	0.06	0.18	0.35	0.14	0.08	0.06	0.08	0.19
Max.	0.2	0.6	37.0	57.5	0.4	1.2	0.4	2.1	1.9
Min.	0.1	0.5	36.7	56.8	0.1	1.1	0.3	1.9	1.5

10. Crucible from Casa da Moeda, Porto

Table 31. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrix of a crucible from the Porto Mint.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO
Porto CI/91/3032/60	**0.2**	**0.7**	**36.6**	**56.9**	**0.1**	**1.4**	**0.3**	**1.8**	**2.0**
Std. dev.	0.05	0.15	0.35	0.55	0.08	0.15	0.09	0.20	0.14
Max.	0.2	0.9	36.8	57.5	0.2	1.6	0.4	2.0	2.2
Min.	0.1	0.5	36.1	56.2	0.0	1.3	0.2	1.6	1.8

11. Crucible from Weyerstraße, Cologne

Table 32. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrix of a crucible from Cologne.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	FeO
D-94/4/s1	**0.5**	**0.5**	**25.2**	**65.3**	**0.5**	**0.5**	**3.0**	**0.8**	**1.7**	**2.1**
Std. dev.	0.13	0.18	0.34	0.54	0.06	0.05	0.14	0.22	0.11	0.16
Max.	0.6	0.8	25.6	65.9	0.5	0.6	3.2	1.0	1.8	2.3
Min.	0.3	0.4	24.8	64.8	0.4	0.5	2.9	0.6	1.6	1.9

12. Crucibles from Burgsteinfurt

Table 33. Chemical composition by SEM-EDS, normalised to 100 wt%, of the ceramic matrices of crucibles from Burgsteinfurt.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	FeO	CuO	ZnO
BGF 01	**0.2**	**0.5**	**36.7**	**56.3**	**0.1**	**1.4**	**0.5**	**2.0**	**1.9**	**≤0.1**	**0.3**
Std. dev.	0.09	0.10	0.20	0.30	0.13	0.26	0.02	0.06	0.17	0.08	0.22
Max.	0.3	0.6	36.9	56.8	0.3	1.8	0.5	2.1	2.2	0.1	0.7
Min.	0.1	0.4	36.4	56.0	0.0	1.1	0.5	2.0	1.8	0.0	0.2
BGF 02	**0.1**	**0.5**	**36.3**	**57.0**	**0.3**	**1.7**	**0.4**	**2.0**	**1.6**	**-**	**0.3**
Std. dev.	0.04	0.03	0.47	0.37	0.08	0.17	0.02	0.13	0.14	-	0.30
Max.	0.2	0.5	36.7	57.5	0.4	1.9	0.4	2.1	1.8	-	0.7
Min.	0.1	0.4	35.6	56.6	0.2	1.5	0.4	1.8	1.4	-	0.0
BGF 03	**0.2**	**0.5**	**37.6**	**56.0**	**0.3**	**1.6**	**0.4**	**1.8**	**1.5**	**-**	**0.1**
Std. dev.	0.11	0.04	0.11	0.33	0.09	0.28	0.08	0.07	0.11	-	0.07
Max.	0.3	0.5	37.7	56.3	0.4	2.1	0.6	1.9	1.7	-	0.3
Min.	0.1	0.4	37.5	55.5	0.2	1.4	0.3	1.8	1.4	-	0.0

Appendix 2. Analytical details of metallurgical residues

Note:
This Appendix is presented for documentation purposes only, to illustrate the range of metallurgical/chymical activities carried out in the technical ceramics, and to provide supportive analytical data for the few metallurgical residues discussed within the text. The more comprehensive discussion and interpretation of the specifics of each metallurgical reaction, and of the relevant technological contexts, fall beyond the scope of this thesis, and are therefore left out for future work.

1. Oberstockstall

Crucible OB 494

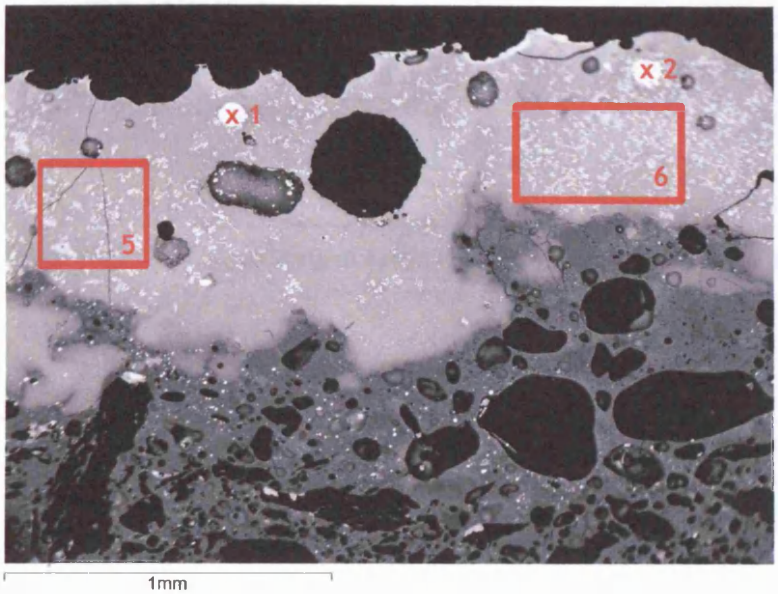


Figure 192. Interface between the crucible and the adhering slag (OB 494/s1, BSE, 50x). SEM-EDS analytical results for the spots and areas marked are given in the table below.

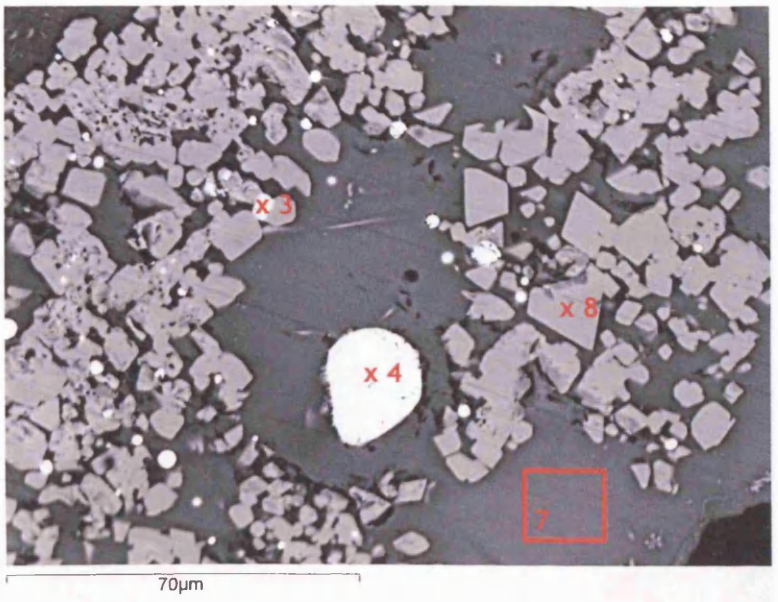


Figure 193. Detail of the slag (OB 494/s1, BSE, 800x). Analytical results for the spots and areas marked are given in the table below.

	S	Fe	Cu	As	Sb	Pb
1	1.1	5.3	23.4	-	70.3	-
2	21.2	5.7	71.8	-	1.3	-
3	0.3	-	10.6	0.4	84.0	4.6
4	2.7	-	78.5	-	18.9	-

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	Cl	K_2O	CaO	TiO_2	FeO	CuO	As_2O_3	Sb_2O_3	PbO
5	3.7	1.9	10.4	29.7	0.8	0.5	0.2	3.1	8.7	0.7	32.0	0.6	0.1	7.3	0.4
6	3.5	1.8	10.6	30.9	1.0	0.3	0.4	3.2	9.1	0.8	31.1	0.3	-	6.6	0.4
7	4.6	2.2	10.7	40.1	1.6	0.7	0.3	5.0	12.5	-	21.2	-	-	-	-
8	-	1.6	2.8	-	-	-	-	-	-	0.4	94.0	-	1.1	-	-

Table 34. SEM-EDS analytical results for the slag within the crucible OB 494.

Scorifier OB 269

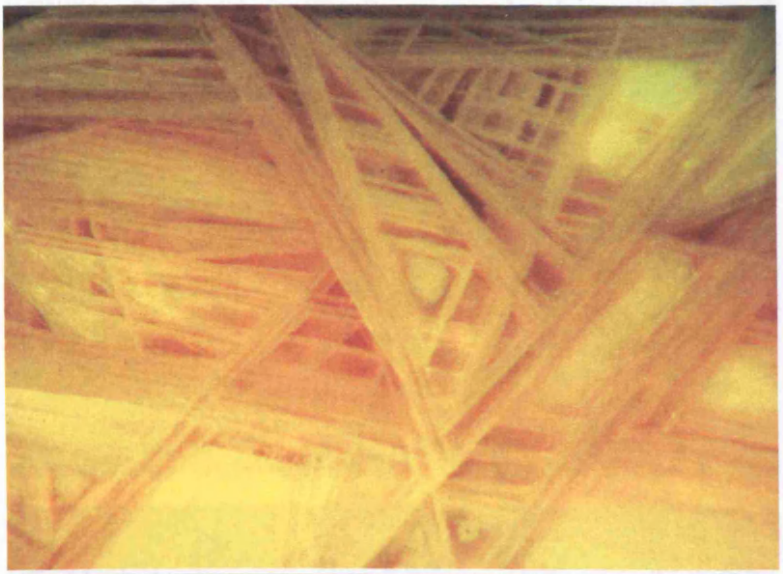


Figure 194. Slag on top of scorifier OB 269 (OB 269/s1, XPL, 50x, long axis represents ~1 mm).

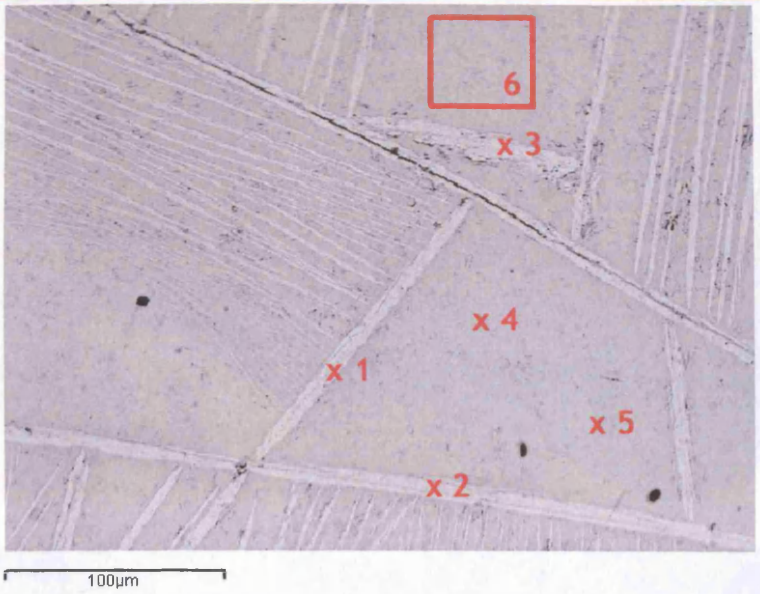


Figure 195. Detail of the same slag under higher magnification (OB 269/s1; BSE, 800x). SEM-EDS analytical results for the marked areas are given in the table below.

	Al_2O_3	SiO_2	CaO	FeO	PbO
1	0.4	1.6	0.5	0.3	97.2
2	0.6	2.2	0.4	0.4	96.4
3	0.2	0.5	-	-	99.4
4	2.7	8.3	0.5	0.6	88.0
5	2.6	8.5	0.5	0.4	87.9
6	2.8	8.8	0.5	0.7	87.2

Table 35. SEM-EDS analytical results for the slag within the scorifier OB 269.

Scorifier 282

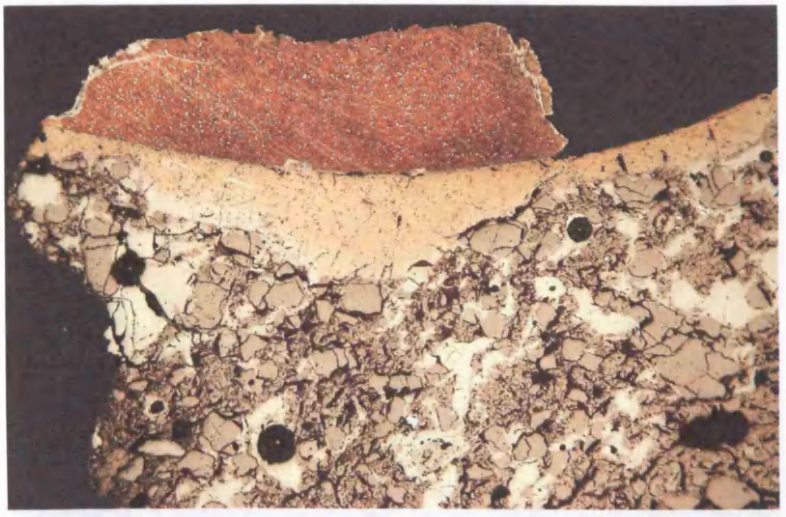


Figure 196. Interface between the fabric of scorifier OB 282 and the residues adhering to the inner surface (top) (OB 282/s1, PL, 50x, long axis represents ~2 mm).

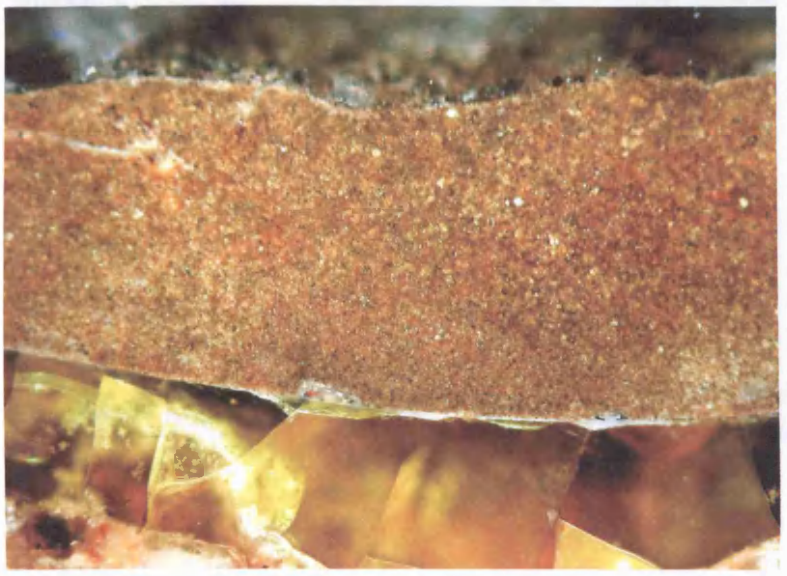


Figure 197. Detail of the lead prill (top) sitting on a layer of lead silicate slag (OB 282/s1, XPL, 100x, long axis represents ~1 mm).

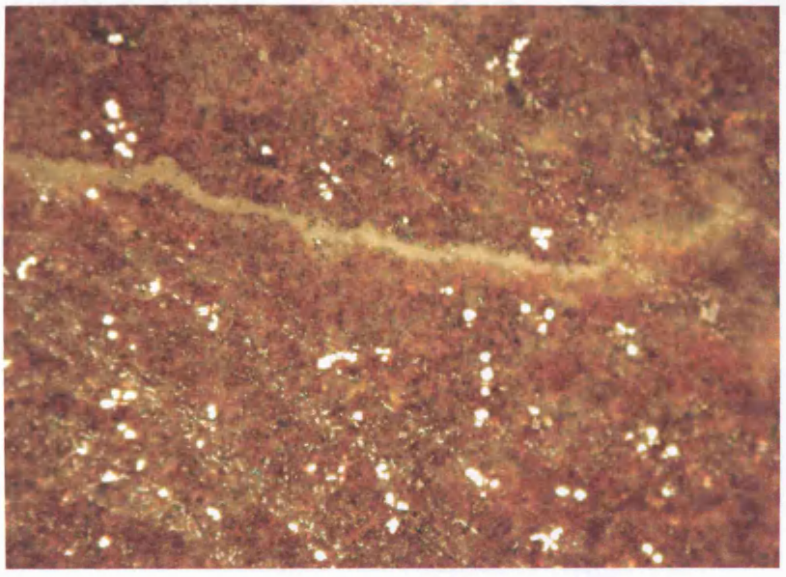


Figure 198. Bottom-left, close-up of the lead prill, showing bright globules with a high silver concentration (OB 282/s1, PL, 500x, long axis represents ~200 µm).

	Ni	Cu	Ag	Pb
1	-	52.9	35.5	11.7
2	-	48.1	34.1	17.8
3	-	43.4	26.5	30.1
4	0.5	45.1	38.2	16.2
5	-	-	6.6	93.4
6	-	-	1.6	98.4

Table 36. Chemical composition by SEM-EDS of some of the bright globules in the image (1-4) and the metallic matrix (5-6). Note that ~5 wt% oxygen from the lead matrix was neglected.

Scorifier 284

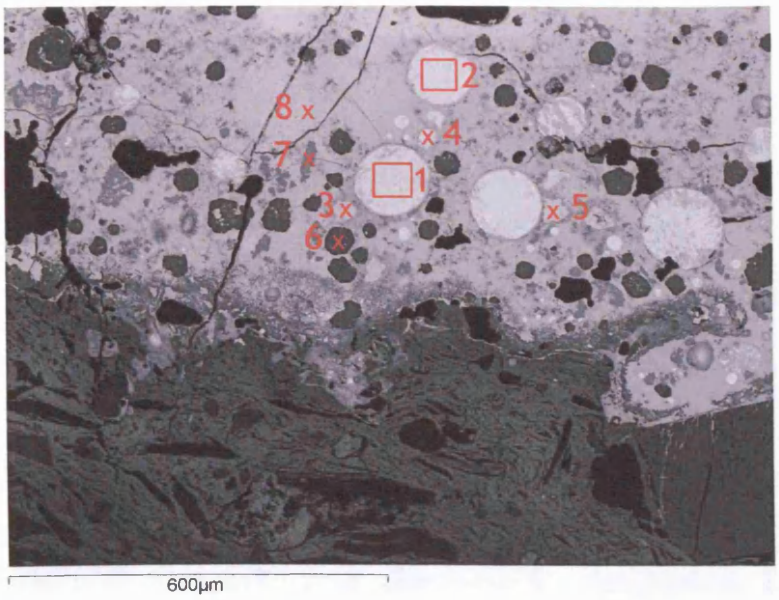


Figure 199. Interface between the ceramic body and the slag in scorifier OB 284. (OB 284/s1, BSE, 50x). SEM-EDS analytical results for the spots and areas marked are given in the table below.

	O	Cl	Cu	As	Sb	Pb
1	1.6	-	1.1	1.0	75.9	20.4
2	2.1	-	1.8	-	68.0	28.2
3	13.7	-	-	-	86.3	-
4	4.7	0.8	-	1.0	86.1	7.5
5	13.9	-	-	-	86.1	-

	MgO	Al_2O_3	SiO_2	P_2O_5	SO_2	K_2O	CaO	FeO	Sb_2O_3	PbO
6	-	17.1	42.4	8.8	-	15.2	12.9	1.4	2.2	-
7	2.6	1.6	-	-	1.6	0.5	-	90.7	3.0	-
8	-	2.3	6.4	-	-	-	2.8	5.1	64.1	19.3

Table 37. SEM-EDS analytical results for the slag within the scorifier OB 284 (I).

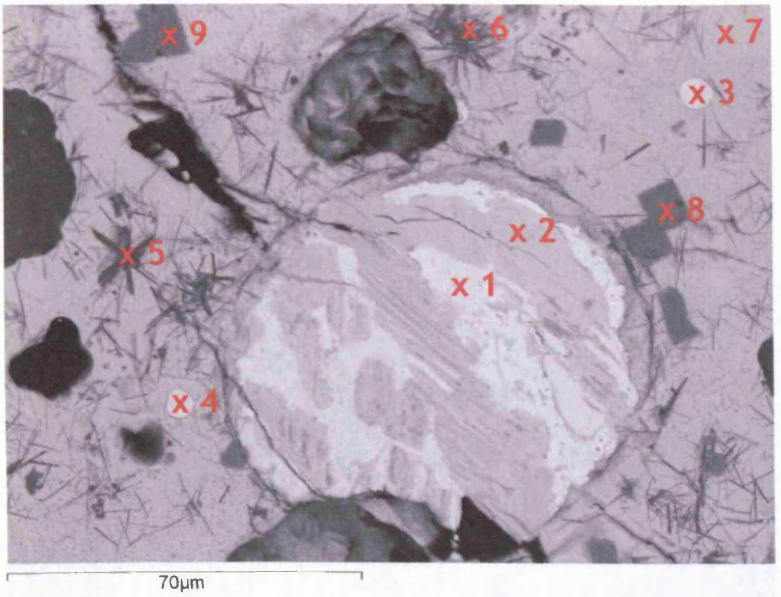


Figure 200. Detail of the slag within scorifier OB 284 (OB 284/s1, BSE, 800x). SEM-EDS analytical results for the spots marked are given in the table below.

	O	Cl	As	Sb	Pb
1	4.7	4.9	-	15.9	74.5
2	12.6	-	-	84.6	2.8
3	-	-	0.8	95.2	4.1
4	-	-	0.3	96.2	3.5

	MgO	Al_2O_3	SiO_2	P_2O_5	CaO	TiO_2	FeO	As_2O_3	Sb_2O_3	PbO
5	8.0	8.0	41.0	2.1	23.4	2.1	14.4	-	-	1.3
6	7.2	7.5	37.9	0.2	21.9	2.0	12.8	-	9.7	0.8
7	-	1.9	5.3	-	3.7	-	5.5	-	74.5	9.2
8	0.8	2.3	-	-	-	0.8	93.3	-	2.8	-
9	2.5	3.0	-	-	-	2.0	91.8	-	0.8	-

Table 38. SEM-EDS analytical results for the slag within the scorifier OB 284 (II).

2. Old Ashmolean laboratory, Oxford

Crucible OX 1413

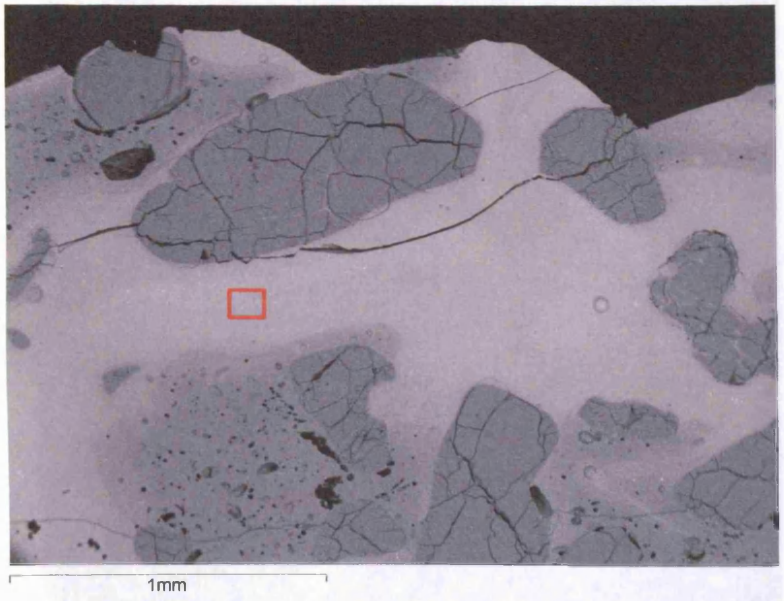


Figure 201. Detail of the chemical attack into the fabric of the crucible from the inner surface (top) (OX 1413/s1, BSE, 50x). The area marked appears magnified in the next figure.

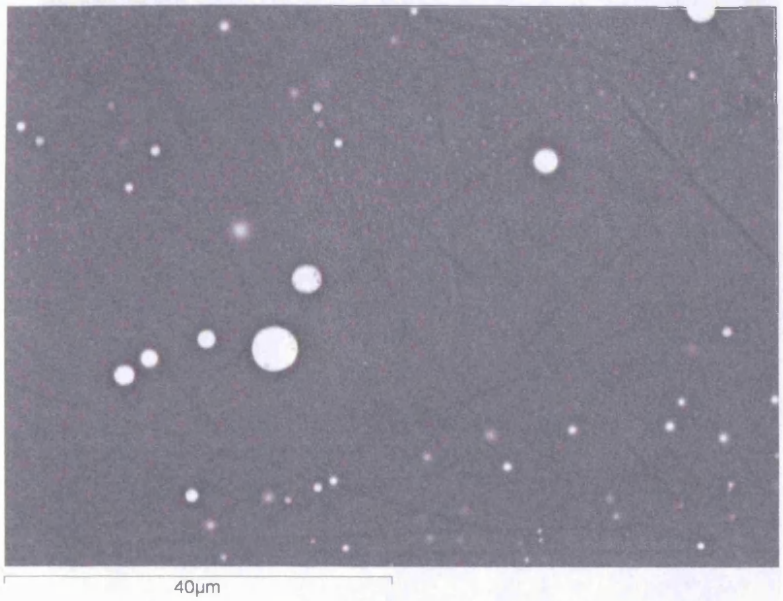


Figure 202. Detail of the slag within the fabric of the crucible (OX 1413/s1, BSE, 1500x). SEM-EDS analytical details are given in the table below.

	S	Mn	Fe	Zn
1	35.8	57.1	5.9	1.3
2	33.2	60.8	5.5	0.5
3	34.5	58.7	5.6	1.2

	Al_2O_3	SiO_2	SO_2	K_2O	CaO	TiO_2	MnO	FeO	ZnO
4	21.0	49.1	2.2	1.0	0.4	1.0	23.9	1.1	0.3

Table 39. SEM-EDS analytical results for the slag within the crucible OX 1413. Rows 1-3 present the composition for three of the bright globules in the previous image. Row 4 presents the composition of the glassy matrix.

3. Cripplegate Buildings, London

Ceramic sample WFG 118/107

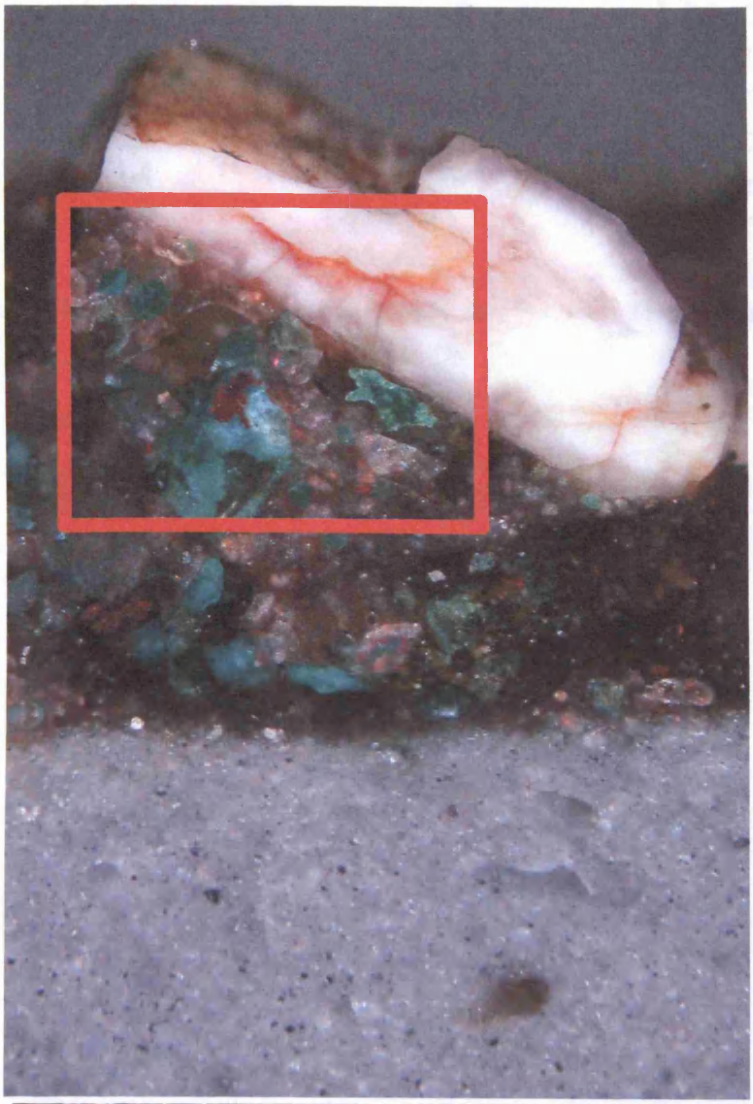


Figure 203. Detail of the inner surface of a 'stoneware crucible' from Cripplegate (WFG 118/107/s2, XPL, 50x, long axis represents ~2 mm). The area marked appears magnified in the next figure.

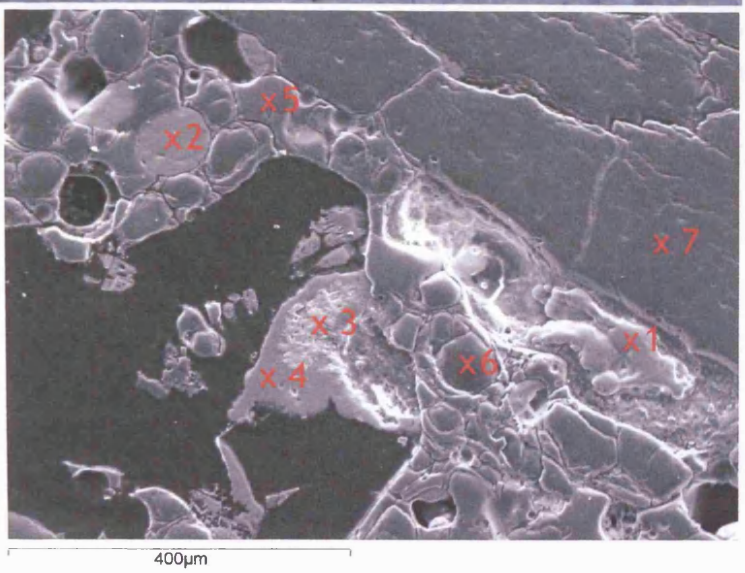


Figure 204. Detail of the sediment adhering to the inner surface (WFG 118/107/s2, BSE, 140x). SEM-EDS analytical results are given in the table below.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	FeO	CuO	ZnO
1	-	-	-	-	2.6	-	-	1.2	-	1.4	93.6	1.2
2	-	-	-	-	1.3	-	-	0.8	-	-	96.2	1.8
3	-	-	-	-	-	1.0	-	-	-	-	99.0	-
4	-	-	-	-	1.3	-	-	0.8	-	-	95.1	2.7
5	1.4	1.1	11.9	65.4	1.1	0.3	4.4	9.1	0.7	3.1	-	1.6
6	-	-	-	99.2	-	-	-	-	-	-	0.8	-
7	-	-	-	100.0	-	-	-	-	-	-	-	-

Table 40. SEM-EDS analytical results for the metallurgical residues within sample WFG 118/107.

4. Jamestown

Crucible JR 124F/C1

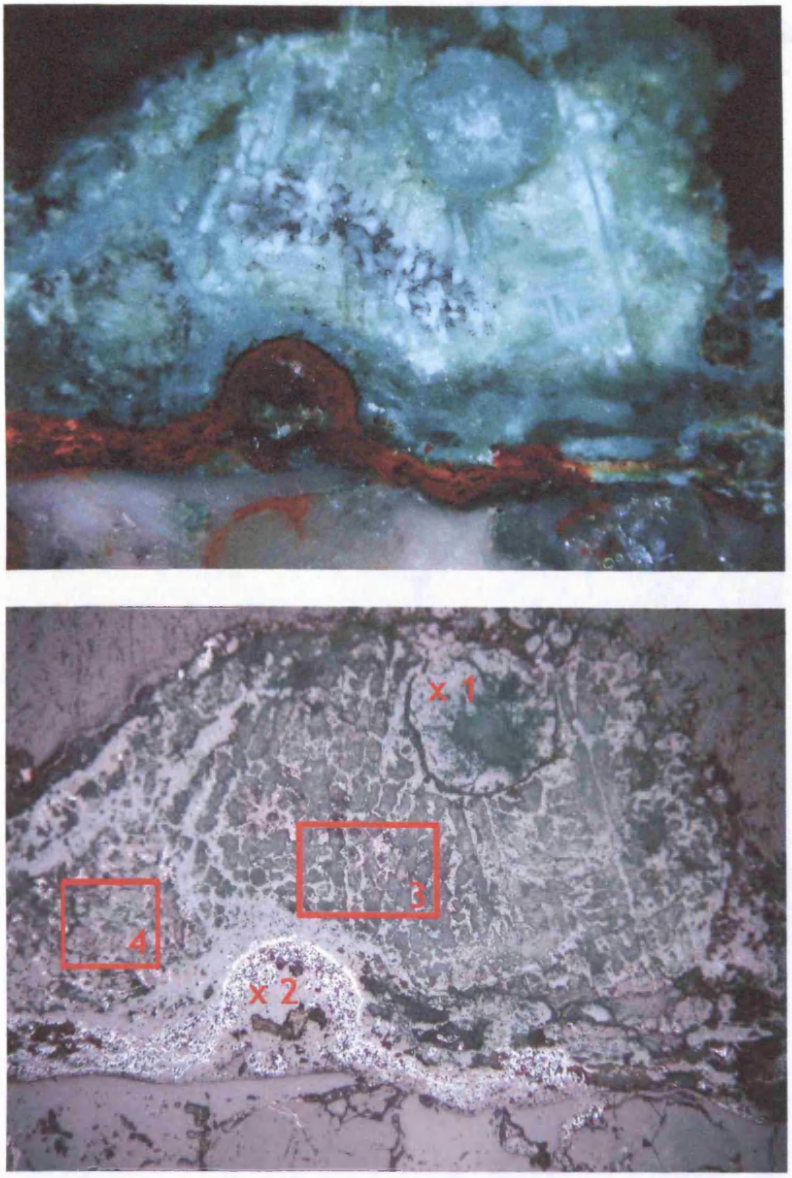


Figure 205. Two views of the section through one of the corroded prills adhering to the inner surface of the crucible. (JR 124F/C1, top XPL, bottom PL, 100x, long axis represents ~1 mm). SEM-EDS analytical results for the spots and areas marked are given in the table below. Spectrum 5 is a large area covering most of the prill. Note that the copper-rich dendrites have been corroded away, therefore the results show unrealistically high tin levels.

	Al_2O_3	SiO_2	P_2O_5	Cl	FeO	CuO	ZnO	SnO_2	PbO
1	-	-	-	-	-	99.9	<0.1	-	-
2	-	-	-	18.4	-	79.1	-	-	2.5
3	-	4.5	1.5	-	2.7	10.6	-	76.2	4.5
4	-	5.5	2.0	-	3.5	10.5	-	74.5	3.9
5	1.1	4.5	1.6	2.5	3.1	32.3	-	50.9	4.0

Table 41. SEM-EDS analytical results for the metallic prill within the crucible JR 124F/C1.

Metallic prill JR 69E/224

Figure 206. Metallic prill JR 69E/224. Its width is ~10 mm.

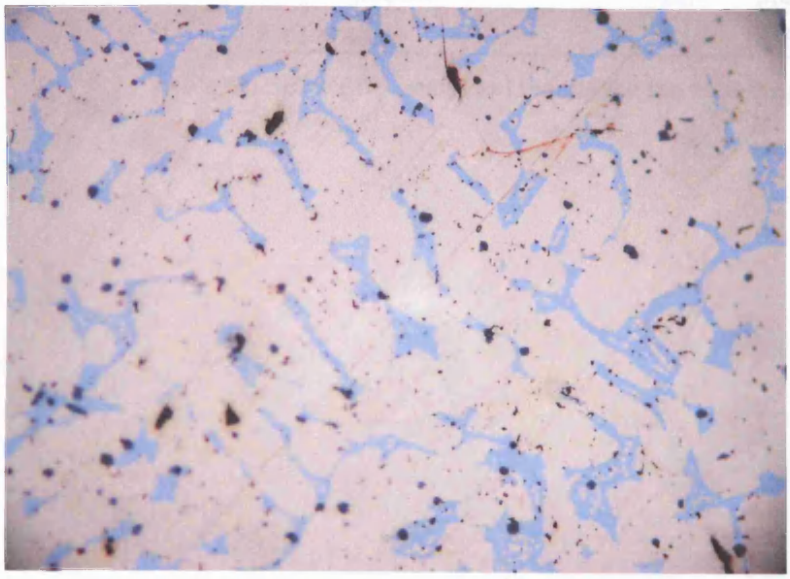


Figure 207. Detail of the microstructure of the prill, showing large dendrites of *alpha* (golden) and interdendritic spaces of *alpha-delta* (grey), as well as discrete globules (dark spots) (JR 69E/224, UPL, 500x, long axis represents ~200 µm).

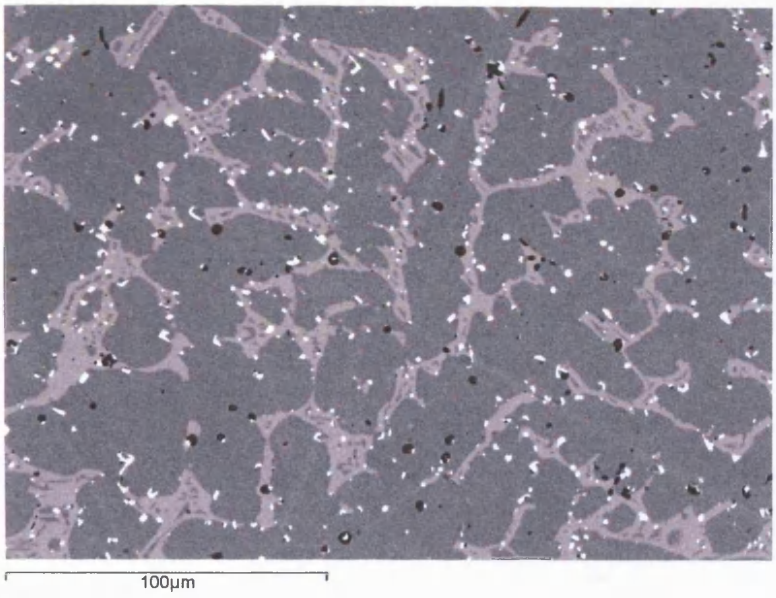


Figure 208. BSE photomicrograph of the same prill. SEM-EDS analytical results are given in the following table (JR 69E/224, 500x).

	S	Fe	Cu	Sn	Pb
Dendrite	-	-	84.8	15.3	-
Dendrite	-	-	85.4	14.7	-
Dendrite	-	-	85.4	14.6	-
Inter-dendrite	-	-	68.0	32.0	-
Inter-dendrite	-	-	68.4	31.6	-
Dark globule	17.8	-	77.0	5.2	-
Dark globule	18.4	1.4	80.2	-	-
Bright globule	-	-	22.7	3.6	73.7
Bright globule	-	-	22.2	3.8	74.0
Full area	-	-	80.1	18.2	1.7

Table 42. SEM-EDS analytical results for the metallic prill JR 69E/224.

Crucible A631/CC2

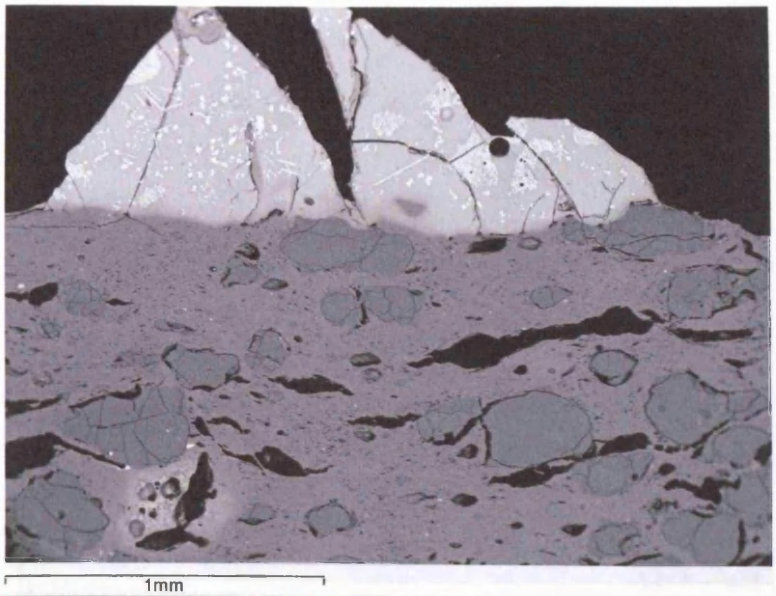


Figure 209. Section through a small triangular crucible, showing the quartz-tempered fabric with one small bloated region (bottom) and slag remains adhering to the inner surface (top) (A631/CC2/s1, BSE, 50x).

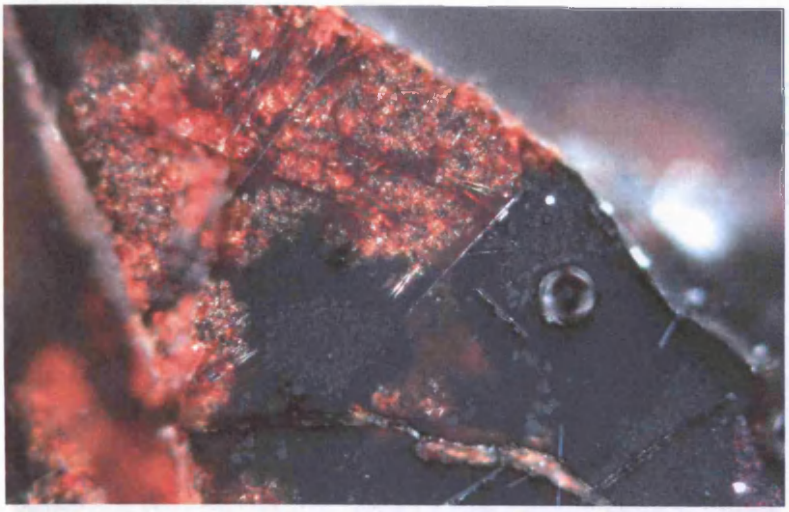
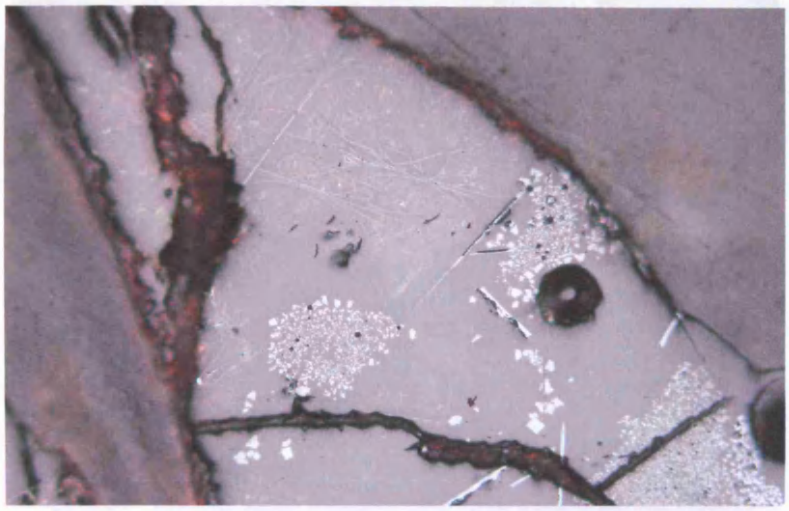


Figure 210. Detail of the slag adhering to the inner surface of the crucible. Note the very glassy appearance and the clustering of bright crystals (A631/CC2/s1, top XPL, bottom PL, 200x, long axis represents ~500 µm).



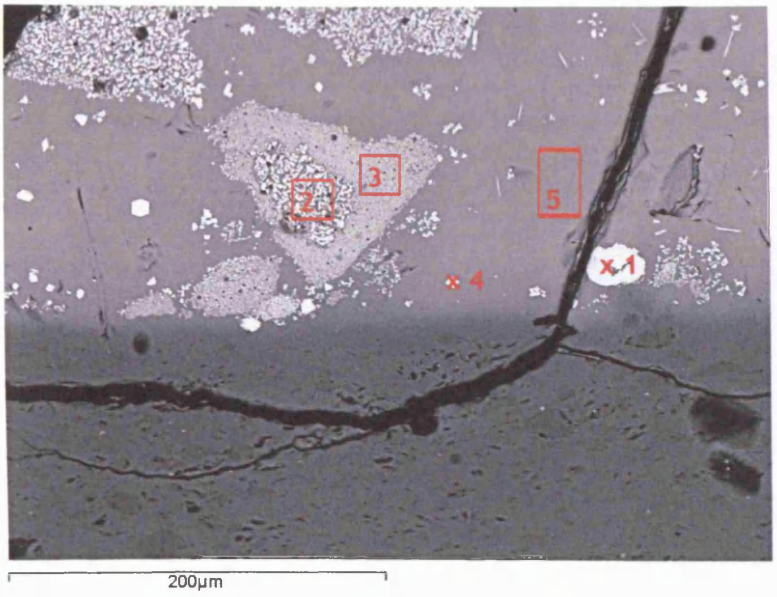


Figure 211. BSE photomicrograph of the interface between the slag and the crucible fabric. SEM-EDS analytical results for the spots and areas marked are given in the following table. Spectrum 6 is a large area of the slag. (A631/CC2/s1, 300x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO	NiO
1	-	0.9	-	-	-	-	-	-	-	0.4	83.2	15.6
2	5.7	-	1.6	19.9	0.4	0.7	3.1	0.2	0.4	-	68.0	-
3	5.5	-	1.5	23.6	0.6	0.9	3.7	-	-	0.5	63.1	0.7
4	-	0.4	-	0.5	-	-	0.3	-	-	0.6	97.0	1.2
5	9.8	-	1.4	46.9	1.1	1.0	7.7	0.7	-	-	31.5	-
6	8.9	-	1.5	40.7	1.1	0.9	6.3	0.5	-	-	40.1	-

Table 43. SEM-EDS analytical results for the slag within the crucible A631/CC2 (I).

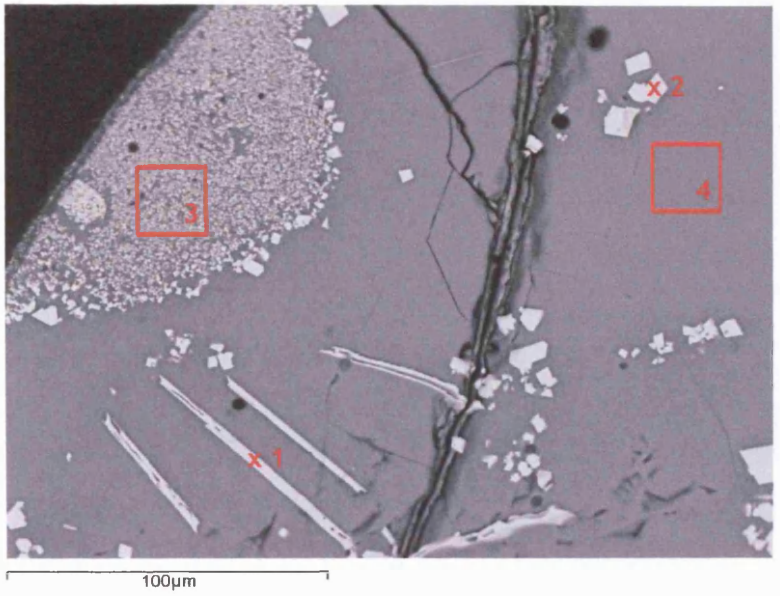


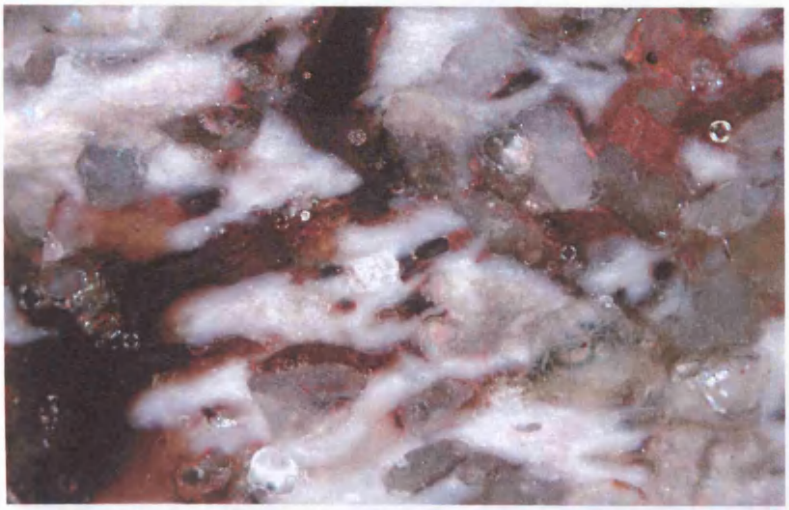
Figure 212. Detail of the same slag. SEM-EDS analytical results for the spots and areas marked are given in the following table (A631/CC2/s1, BSE, 500x).

	Na_2O	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO
1	0.5	-	0.9	-	-	0.2	-	0.7	-	97.8
2	-	-	0.4	-	-	0.2	-	-	0.9	98.5
3	7.5	1.0	27.9	0.7	0.9	4.6	0.7	-	0.4	56.4
4	10.7	1.6	41.8	1.3	1.0	7.4	1.1	0.3	-	34.8

Table 44. SEM-EDS analytical results for the slag within the crucible A631/CC2 (II).

Crucible JR 1024/CC3

Figure 213. Crucible rim fragment JR 1024/CC3. Note the shiny glaze and relatively smooth surface resulting from contamination and vitrification during use



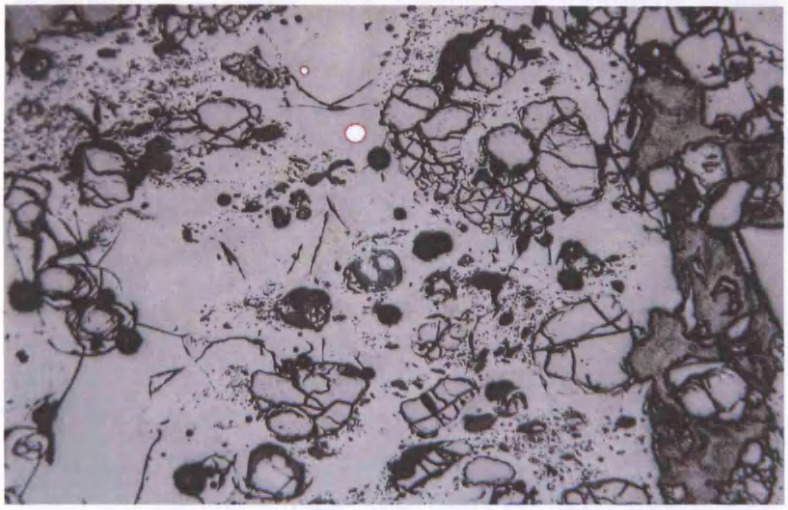


Figure 214. Section through the ceramic fabric of the crucible, showing the intense penetration of glassy slag (top image, brown) containing metallic prills, and the severe shattering of the quartz grains (bottom image) (JR 1024/CC3/s1, top XPL, bottom PL, 50x, long axis represents ~2 mm).

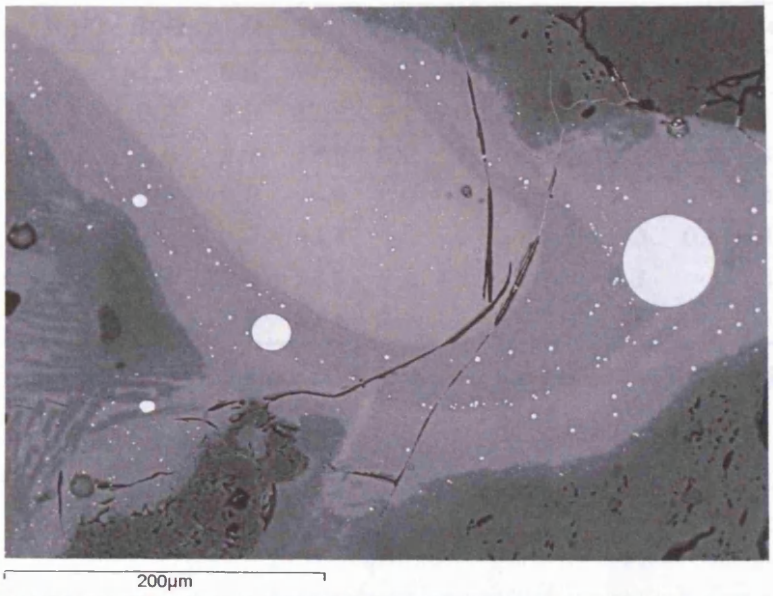


Figure 215. Detail of the slag penetrating into the ceramic fabric, the slag being very rich in lime and copper oxide. Note the abundance of minuscule copper prills (bright) (JR 1024/CC3/s1, BSE, 250x).

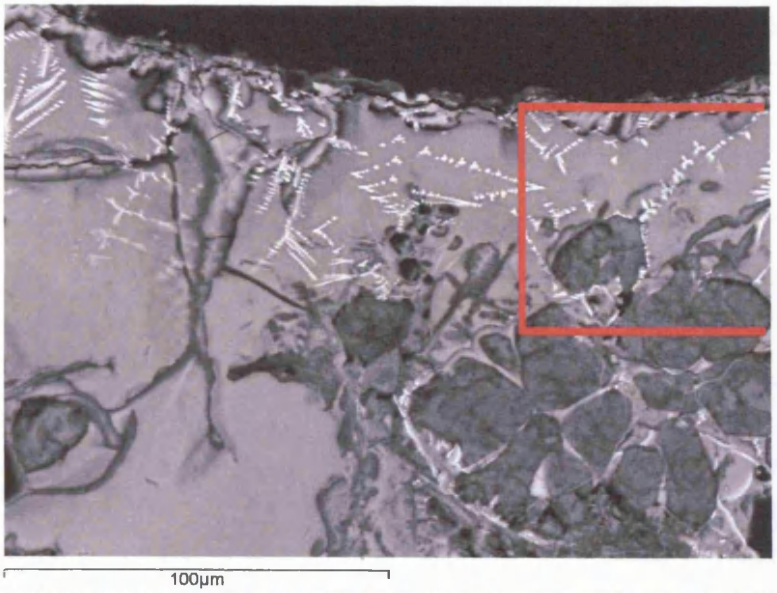


Figure 216. Detail of the slag on the inner surface of the crucible. The marked area is shown in the following figure under higher magnification (JR 1024/CC3/s1, BSE, 600x).

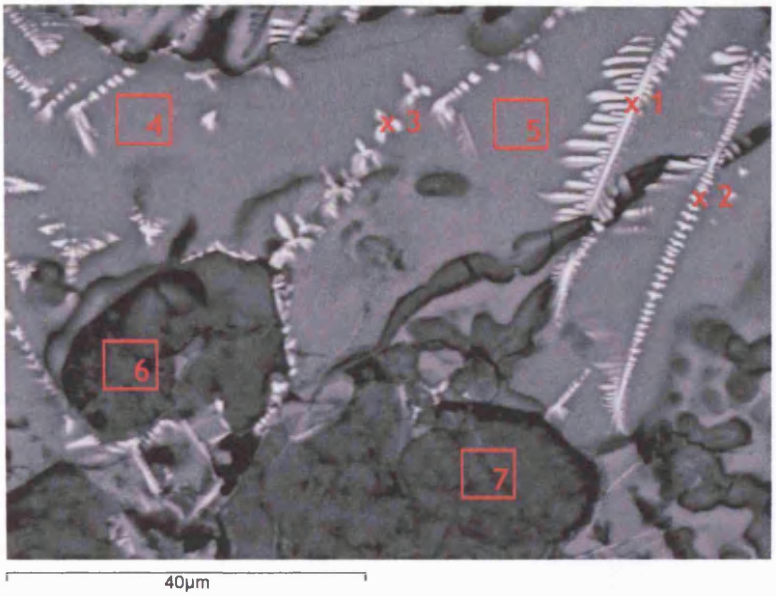


Figure 217. Detail of the slag shown in the previous figure. SEM-EDS analytical results for the spots and areas marked are given in the following table (JR 1024/CC3/s1, BSE, 1400x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO	CuO	ZnO	As_2O_3	PbO
1	-	1.3	6.6	27.7	0.6	0.8	10.1	0.5	0.2	2.0	50.3	-	-	-
2	-	0.7	3.9	17.9	0.2	0.7	5.9	-	-	1.3	69.0	0.4	-	-
3	-	1.6	7.9	37.9	0.7	1.2	15.4	0.7	0.3	3.3	31.1	-	-	-
4	-	2.1	9.0	44.6	0.9	0.8	21.6	1.5	0.5	4.7	14.7	-	-	-
5	-	2.2	9.2	43.7	1.0	0.8	21.8	1.0	0.6	4.7	15.1	-	-	-
6	6.0	0.2	21.8	54.0	3.8	3.5	1.8	0.3	-	3.8	2.6	-	0.2	2.1
7	6.6	-	23.9	57.8	2.5	3.5	0.3	-	-	1.5	1.6	-	-	2.3

Table 45. SEM-EDS analytical results for the slag within the crucible JR 1024/CC3.

5. Burgsteinfurt

Crucible BFG 01

Figure 218. View of the inside of the crucible BGF 01. Note the lumpy texture and the white film of zinc oxide.

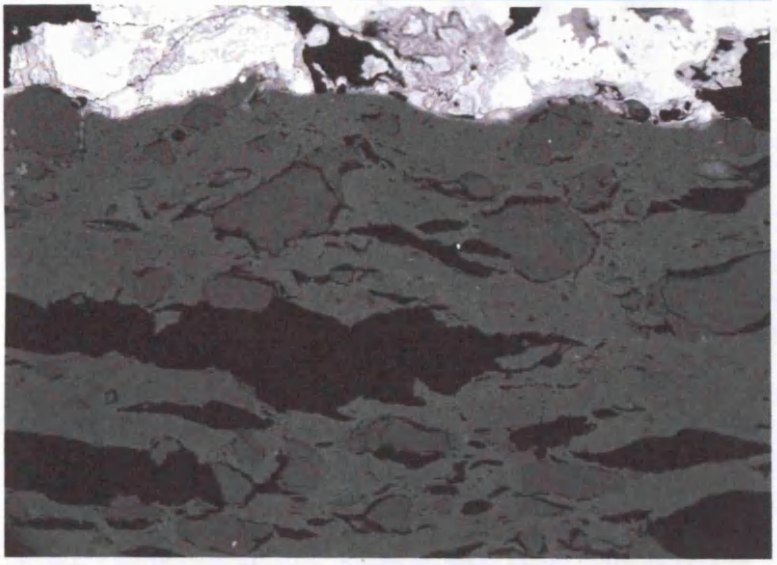


Figure 219. Section through the crucible BGF 01, showing a substantial layer of zinc oxide adhering to the inner surface (BGF 01/s1, BSE, 50x).

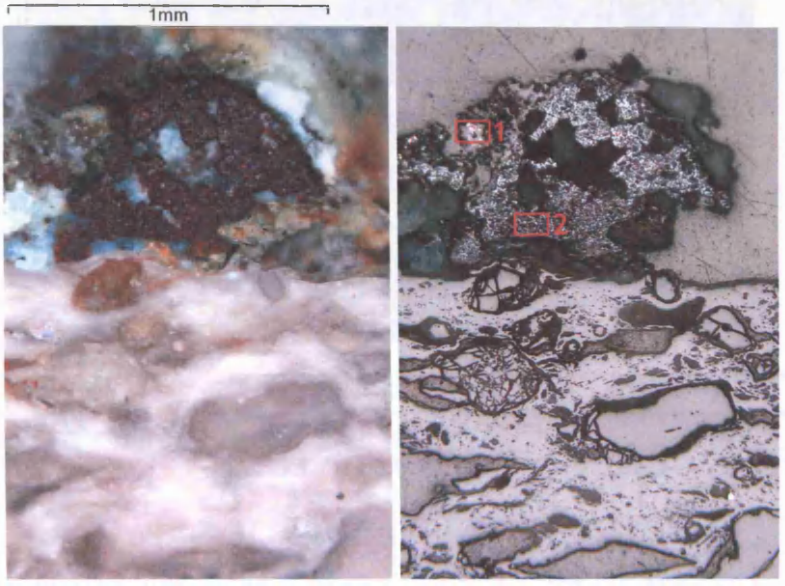


Figure 220. Two views of a corroded metallic prill on the inner surface of the crucible. The areas 1 and 2 marked on the right image are those magnified in the following pictures (BGF 01/s1, left XPL, right PL, 50x, long axis represents ~2 mm).

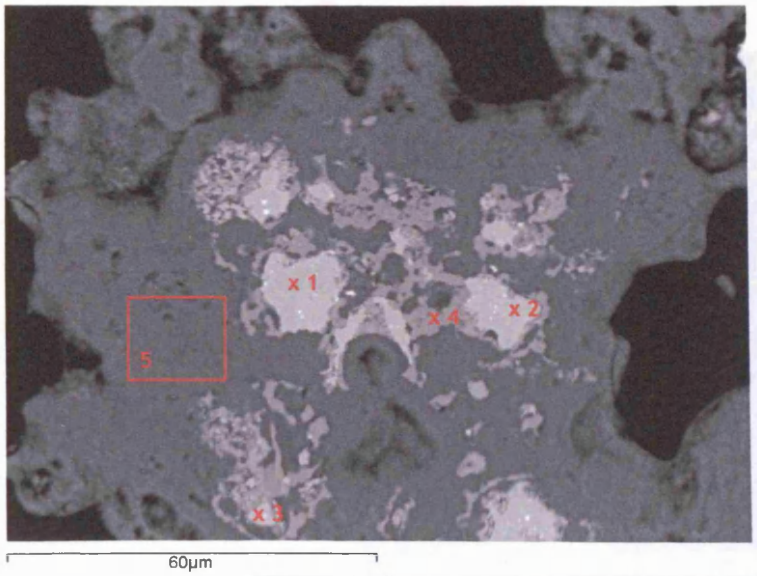


Figure 221. Area 1 as marked in Figure 220. SEM-EDS analytical details are given in the table below (BGF 01/s1, BSE, 1000x).

	Si	S	Ca	Mn	Fe	Cu	Zn	Pb
1	-	-	-	-	0.2	54.1	45.8	-
2	-	-	-	-	0.4	53.4	46.2	-
3	-	23.1	-	0.3	0.5	8.6	67.5	-
	SiO_2	SO_2	CaO	MnO	FeO	CuO	ZnO	PbO
4	-	-	0.3	0.6	1.0	2.5	95.7	-
5	0.9	0.8	0.7	-	1.0	9.0	85.3	2.3

Table 46. SEM-EDS analytical results for the residues within the crucible BGF 01 (I).

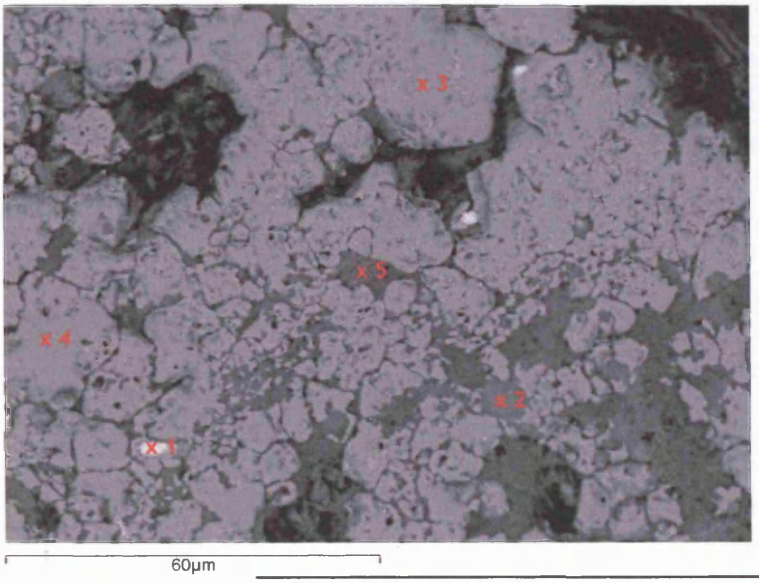


Figure 222. Area 2 as marked in Figure 220. SEM-EDS analytical details are given in the table below (BGF 01/s1, BSE, 1000x).

	O	S	Cl	Cu	Zn	As	Sn	Pb
1	12.3	-	2.9	15.6	-	12.5	-	56.8
2	14.7	0.6	3.7	47.8	13.5	-	19.7	-
3	8.3	-	-	91.7	-	-	-	-
4	8.8	-	-	91.3	-	-	-	-
5	19.9	-	8.9	67.3	1.23	-	-	2.6

Table 47. SEM-EDS analytical results for the residues within the crucible BGF 01 (II).

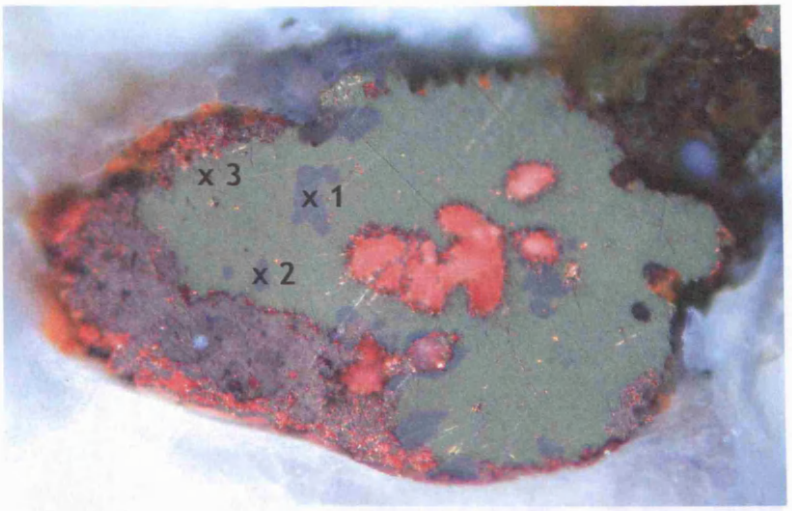


Figure 223. Prill of high-zinc brass identified on the inner surface of the crucible BGF 01. Note the *gamma* brass crystals (grey) within a *beta* brass matrix (golden), and small lead globules. SEM-EDS analytical details are given in the table below. Spectra 4 and 5 correspond to two 30 x 30 µm areas within the metal (BGF 01/s1, XPL, 500x, long axis represents ~200 µm).

	Fe	Cu	Zn	Sn	Pb
1	-	42.2	57.5	0.6	-
2	-	42.6	56.9	0.6	-
3	0.9	18.5	19.4	-	61.4
4	0.3	51.1	48.0	0.1	0.5
5	0.2	48.4	50.3	0.4	0.8

Table 48. SEM-EDS analytical results for the metallurgical residues within the crucible BGF 01 (III).

Crucible BGF 02

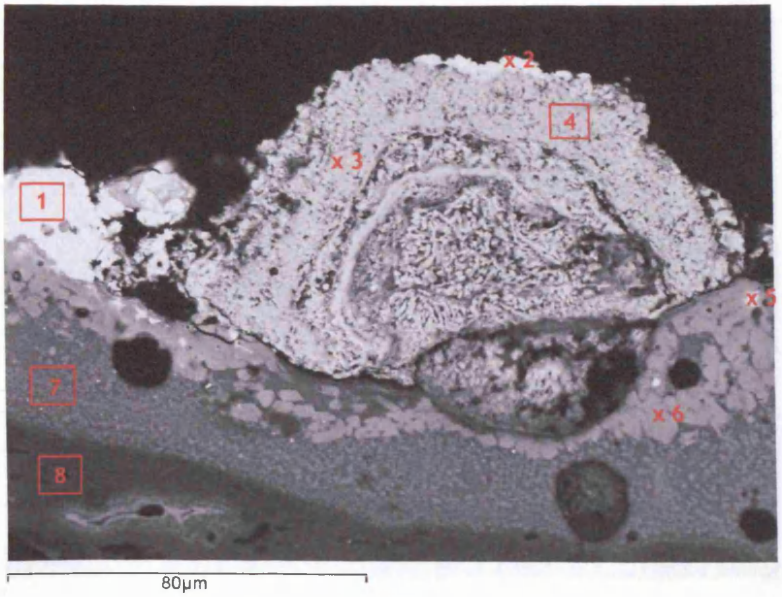


Figure 224. Detail of the inner surface of a crucible, showing a corroded metal prill and other submetallic residues. SEM-EDS analytical results for the spots and areas marked are given in the table below (BGF 02/s2, BSE, 700x).

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	Cl	K_2O	CaO	TiO_2	MnO	FeO	NiO	CuO	ZnO	SnO_2	PbO
1	-	-	-	0.6	-	0.7	-	3.1	-	-	-	-	1.5	2.4	-	91.7
2	-	-	-	-	-	0.8	-	2.1	-	-	-	-	8.1	1.1	-	88.0
3	-	-	-	-	-	-	-	-	-	-	0.3	-	95.7	2.1	1.4	0.7
4	-	-	-	0.7	-	-	-	-	-	-	0.3	0.5	91.8	3.5	2.2	1.1
5	-	-	5.5	28.4	0.8	-	0.7	0.3	0.7	-	6.1	-	6.8	12.3	-	38.4
6	-	0.8	10.5	22.8	-	-	-	0.3	0.4	0.7	12.8	-	0.8	51.0	-	-
7	-	0.6	26.5	26.7	0.9	-	0.8	5.8	1.1	0.7	5.7	-	1.0	30.3	-	-
8	0.5	0.2	35.0	55.6	0.2	-	4.2	-	2.0	-	1.1	-	-	1.2	-	-

Table 49. SEM-EDS analytical results for the crucible BGF 02.

Crucible BGF 03

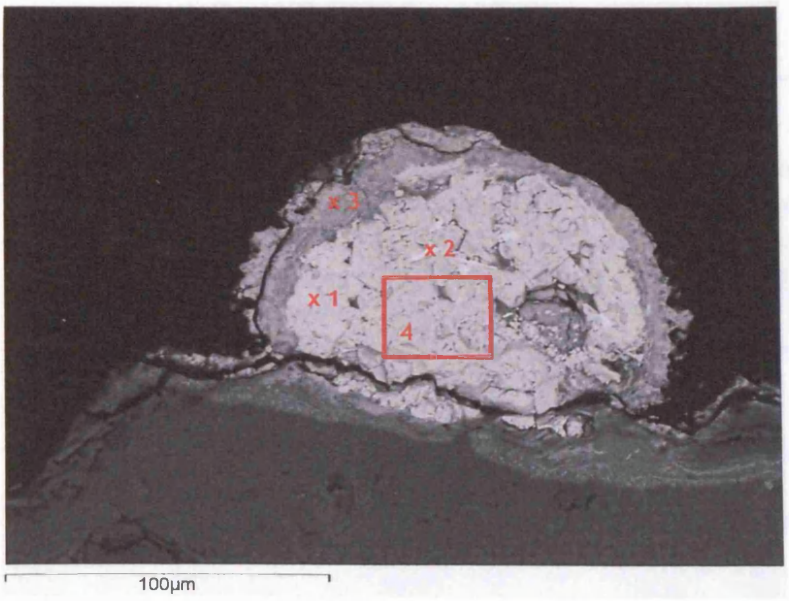


Figure 225. Detail of the inner surface of another crucible, showing another corroded prill. SEM-EDS analytical results for the spots and areas marked are given in the table below (BGF 03/s1, BSE, 500x).

	SiO_2	P_2O_5	SO_2	Cl	CaO	FeO	CuO	ZnO	As_2O_3	SnO_2	PbO
1	-	-	-	5.0	-	-	16.5	-	-	15.5	63.0
2	-	-	-	-	-	-	100.0	-	-	-	-
3	5.7	1.0	1.8	1.2	1.3	1.6	27.5	54.2	0.5	-	5.0
4	-	-	0.5	0.2	-	-	98.4	-	-	0.9	-

Table 50. SEM-EDS analytical results for the metallic prill within the crucible BGF 03.

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